

CHEMISTRY LAB MANUAL

For

(ME Only)

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LENDI INSTITUTE OF ENGINEERING AND TECHNOLOGY (An Autonomous Institution)

(Approved by AICTE, Accredited by NBA & NAAC with 'A' Grade, Recognized Under 2(f), 12(B) by UGC and Permanently Affiliated to JNTUG, Vizianagaram) Jonnada (Village), Denkada (Mandal), Vizianagaram District, Andhra Pradesh– 535 005

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A. General Safety Rules

- 1. Listen to or read instructions carefully before attempting to do anything.
- 2. Wear safety goggles to protect your eyes from chemicals, heated materials, or things that might be able to shatter.
- 3. Notify your teacher if any spills or accidents occur
- 4. After handling chemicals, always wash your hands with soap and water.
- 5. During lab work, keep your hands away from your face.
- 6. Tie back long hair.
- 7. Roll up loose sleeves.
- 8. Know the location of the fire extinguisher, fire blanket, eyewash station, and first aid kit.
- 9. Keep your work area uncluttered. Take to the lab station only what is necessary.
- 10. It is suggested that you wear glasses rather than contact lenses.
- 11. Never put anything into your mouth during a lab experiment.
- 12. Clean up your lab area at the conclusion of the laboratory period.
- 13. Never "horse around" or play practical jokes in the laboratory

B. Glassware Safety

- 1. Chipped or cracked glassware should not be used. Show it to the teacher.
- 2. Broken glassware should not be disposed of in a classroom trashcan. There is a special glass disposal container for it.
- 3. When pouring liquids into glassware, make sure the container you are pouring into is resting on a table at least a hands breadth from the edge.
- 4. Pour down a glass stirring rod to prevent liquids from splattering.
- 5. If a piece of glassware gets broken, do not try to clean it up by yourself. Notify the teacher.
- 6. When inserting glass tubing into a rubber stopper, apply a lubricant to the glass and use a twisting motion
- 7. To cut glass tubing, first lay the tube on the lab table and make a scratch in it with a file. Then pick up the tube with the scratch facing away from you, put your thumbs together on the opposite side as the scratch, and break the tube with both hands.
- 8. If you cut glass tubing, always fire polishes the broken ends to remove jagged edges.
- 9. Do not place hot glassware in water. Rapid cooling may make it shatter

C. Chemical Safety

- 1. Wear protective goggles and a lab apron whenever heating or pouring hazardous chemicals.
- 2. Never mix chemicals together unless you are told to do so (and then only in the manner specified).

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- 3. Never taste any chemicals (you should never taste anything in the lab).
- If you need to smell the odor of a chemical, waft the fumes toward your nose with one hand.
 Do not put your nose over the container and inhale the fumes.
- 5. Never pour water into a concentrated acid. Acid should be poured slowly into water.
- 6. Follow the instructions of your teacher when disposing of all chemicals.
- 7. Wash your hands after handling hazardous chemicals.

E. Electrical Safety

- 1. Lay electrical cords where no one can trip on them or get caught in them.
- 2. Be sure your hands and your lab area are dry before using electrical equipment.
- 3. Never poke anything into electrical outlets.
- 4. Unplug cords by pulling the plug and not the cord.
- 5. Unplug all electrical equipment at the end of the lab period.

F. Heating Safety

- 1. Let burners and hotplates cool down before touching them. Test to see if they are cool enough by bringing the back of your hand close to them.
- 2. Use tongs and/or protective gloves to handle hot objects.
- 3. Never reach across an open flame or burner.
- 4. The only type of glassware that may safely be heated is either Kimax or Pyrex.
- 5. Always point the top ends of test tubes that are being heated away from people.
- 6. When heating a test tube, move it around slowly over the flame to distribute the heat evenly.
- 7. Only glassware that is thoroughly dry should be heated.
- 8. Heat glassware by placing it on a wire gauze platform on a ring stand. Do not hold it in your Hand.
- 9. When lighting a burner, wait until a match is struck or the striker is in place before you turn on the gas.
- 10. The amount of air can be adjusted by the air supply valve below the tube of the burner. This regulates the flame temperature and color.
- 11. Never leave a burner or hotplate unattended.

First Aid

Injury: Burns

To do: Immediately flush with cold water until burning sensation is lessened.

Injury: Cuts, bruises

To do: Do not touch an open wound without safety gloves. Pressing directly on minor cuts will stop bleeding in a few minutes. Apply cold compress to bruises to reduce swelling.

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Injury: Fainting

To do: Provide fresh air and have the person recline so that their head is lower than the rest of their body

Injury: The eyes

To do: Flush eyes immediately with plenty of water for several minutes. If a foreign object is lodged in the eye, do not allow the eye to be rubbed.

Injury: Poisoning

To do: Find out what substance was responsible for the poisoning and alert the teacher immediately.

Injury: Spills on the skin

To do: Flush with large quantities of water. For acid spills apply baking soda solution. For base spills apply vinegar or boric acid.

Injury: Electrical shock

To do: Shut off the current at the source. Remove wire with rubber gloves. Alert the teacher immediately.



VISION & MISSION OF THE INSTITUTE

Vision of the Institute

• Producing globally competent and quality technocrats with human values for the holistic needs of industry and society.

Mission of the Institute

- Creating an outstanding infrastructure and platform for enhancement of skills, knowledge and behavior of students towards employment and higher studies.
- Providing a healthy environment for research, development and entrepreneurship, to meet the expectations of industry and society.
- Transforming the graduates to contribute to the socio-economic development and welfare of the society through value based education.



VISION & MISSION OF THE ME DEPARTMENT

Vision of the Department

• Envisions mechanical engineers of highly competent and skilled professionals to meet the needs of the modern society.

Mission of the Department

- Providing a conducive and inspiring learning environment to become competent engineers.
- Providing additional skills and training to meet the current and future needs of the Industry.
- Providing an unique environment towards entrepreneurship by fostering innovation, creativity, freedom and empowerment.



PROGRAM OUTCOMES (POs)

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Program Outcomes:

Engineering Graduates will be able to:

- 1. **Engineering knowledge**: Apply the knowledge of mathematics, science, engineering fundamentals, and an engineering specialization to the solution of complex engineering problems.
- 2. **Problem analysis**: Identify, formulate, review research literature, and analyze complex engineering problems reaching substantiated conclusions using first principles of mathematics, natural sciences, and engineering sciences.
- 3. **Design/development of solutions**: Design solutions for complex engineering problems and design system components or processes that meet the specified needs with appropriate consideration for the public health and safety, and the cultural, societal, and environmental considerations.
- 4. **Conduct investigations of complex problems**: Use research-based knowledge and research methods including design of experiments, analysis and interpretation of data, and synthesis of the information to provide valid conclusions.
- 5. **Modern tool usage**: Create, select, and apply appropriate techniques, resources, and modern engineering and IT tools including prediction and modeling to complex engineering activities with an understanding of the limitations.
- 6. **The engineer and society**: Apply reasoning informed by the contextual knowledge to assess societal, health, safety, legal and cultural issues and the consequent responsibilities relevant to the professional engineering practice.
- 7. Environment and sustainability: Understand the impact of the professional engineering solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
- 8. **Ethics**: Apply ethical principles and commit to professional ethics and responsibilities and norms of the engineering practice.
- 9. **Individual and team work**: Function effectively as an individual, and as a member or leader in diverse teams, and in multidisciplinary settings.
- 10. **Communication**: Communicate effectively on complex engineering activities with the engineering community and with society at large, such as, being able to comprehend and write effective reports and design documentation, make effective presentations, and give and receive clear instructions.



- 11. **Project management and finance**: Demonstrate knowledge and understanding of the engineering and management principles and apply these to one's own work, as a member and leader in a team, to manage projects and in multidisciplinary environments.
- 12. Life-long learning: Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change.



PROGRAM SPECIFIC OUT COMES (PSOs)

ME PSOs

PSO1: Capable of design, develop and implement sustainable mechanical and environmental systems.

PSO2: Qualify in national and international competitive examinations for successful higher studies and employment.



ENGINEERING CHEMISTRY LAB SYLLABUS



ENGINEERING CHEMISTRY LAB (For ME Only)

I YEAR –I SEMESTER SYLLABUS

COURSE OBJECTIVES

Course Objectives:

• To verify the fundamental concepts with experiments

Course Outcomes: At the end of the course, the students will be able to

- CO1: Determine the cell constant and conductance of solutions.
- CO2: Prepare advanced polymer materials.
- CO3: Determine the physical properties like surface tension, adsorption and viscosity.
- CO4: Estimate the viscosity index of lubricating oil.
- CO5: Calculate the hardness of water.

List of Experiments:

- 1. Preparation of a polymer (Bakelite)
- 2. Determination of Hardness of a groundwater sample.
- 3. Determination of Strength of an acid in Pb-Acid battery
- 4. Determination of acid number of lubricating oil
- 5. Determination of Viscosity of lubricating oil by Redwood Viscometer 1/ Redwood Viscometer 2
- 6. Determination of flash and fire points of lubricating oil by Clevelands apparatus
- 7. Determination of the amount of acidity of a given water sample
- 8. Determination of the amount of alkalinity of a given water sample
- 9. Determination of amount of copper in a given sample.
- 10. Determination of amount of zinc in a given sample.
- 11. Estimation of Ferrous Iron by Dichrometry
- 12. Determination of KMnO₄ by using standard oxalic acid solution.
- 13. Estimation of Calcium in port land Cement

Virtual Labs

- 14. Preparation of nanomaterials by precipitation method.
- 15. Adsorption of acetic acid by charcoal
- 16. Determination of percentage Moisture content in a coal sample
- 17. Determination of percentage of Iron in Cement sample by colorimetry
- 18. Determination of Calorific value of gases by Junker's gas Calorimeter

Reference:

• "Vogel's Quantitative Chemical Analysis 6th Edition 6th Edition" Pearson Publications by J. Mendham, R.C. Denney, J.D. Barnes and B. Sivasankar

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6	Determination of Viscosity of lubricating oil by Redwood Viscometer 1/ Redwood Viscometer 2		
7	Determination of flash and fire points of lubricating oil by Clevelands apparatus		
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PRINCIPLES FOR SAFETY IN THE CHEMICAL LABORATORY

Safe practices in the chemical laboratory are of prime importance. A student should consider it an essential part of his or her educational experience to develop safe and efficient methods of operation in a lab. To do this, one must acquire a basic knowledge of properties of materials present in the lab, and one should realize the types of hazards that exist and the accidents and injuries that can result from ignorance or irresponsibility on the part of the student or a neighbor.

Regulations

1. Report all accidents to the instructor or lab assistant immediately.

2. NEVER eat, drink, chew, or smoke in the laboratory.

3. NEVER leave an experiment unattended. Inform the lab assistant if you must leave the lab.

4. After the experiment is completed, turn all equipment off, making sure it is properly stored, and clean your area. Failure to comply with these regulations is cause for immediate dismissal from lab.

Precautions

- 1. Approach the laboratory with a serious awareness of personal responsibility and consideration for others in the lab.
- 2. Become familiar with the location of safety equipment, such as acid-base neutralizing agents, eye wash, fire extinguisher, emergency shower, and fire blanket.
- 3. Pay strict attention to all instructions presented by the instructor. If something is not clear, do not hesitate to ask the instructor or lab assistant.
- 4. Clean up all chemical spills immediately.
- 5. Be aware of all activities occurring within a reasonable proximity of yourself since you are always subject to the actions of others.
- 6. To avoid contamination of community supplies, do not use personal equipment such as spatulas in shared chemicals and replace all lids after use.
- 7. Avoid unnecessary physical contact with chemicals; their toxic properties may result in skin irritation.
- 8. Use all electrical and heating equipment carefully to prevent shocks and burns.
- 9. NEVER handle broken glassware with your hands; use a broom and a dust pan.
- 10. Wash your hands at the end of the laboratory.

Personal Attire

Choice of clothing for the laboratory is mainly left to the discretion of the student. Because of the corrosive nature of chemicals, it is in your best interest to wear comfortable, practical clothing. Long, floppy sleeves can easily come into contact with chemicals.

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A lab coat is suggested to help keep clothes protected and close to the body. Accessories also need consideration.

Assembling Equipment

Equipment should be assembled in the most secure and convenient manner. Utility clamps are provided to fasten flasks, etc., to the metal grid work located at the center of each bench. This keeps top-heavy or bulky equipment away from the edge where it can be knocked easily off the bench. Consider the safe location of the hot plate. Keep it near the grid work to minimize chances of contact with the body. If the aspirator is being used, locate your apparatus near the sink for convenience.

Handling Glassware

Laboratory glassware is usually fragile, and if it is not properly handled, serious injuries may result do not force glass tubing or thermometers into a rubber stopper. Lubricate the tubing or thermometer with glycerol or water, wrap it in a towel, and gently insert it into the stopper by using pressure in a lengthwise direction while rotating it. Always grasp the tubing near the stopper. When removing the tubing, remember to protect your hands with a towel. If there are difficulties with this procedure, ask for the instructor's assistance. Apparatus that can roll should be placed between two immobile objects away from the edge of the bench. Chipped or broken glassware cannot be used. There are special receptacles near each bench for these waste materials. After the experiment is completed, all glassware should be emptied, rinsed, and cleaned.

Acids and Bases

In this lab sequence, you will come in contact with several acids and bases. As with all chemicals, caution must be taken to prevent contact with the skin. When handling these chemicals, keep hands away from the eyes and face until they have been thoroughly washed. If an acid or base comes in contact with your skin, flush the area with large quantities of clean, cold water. Eyes are extremely sensitive. Use the eye wash provided in the laboratory, or wash with water for at least 10 minutes. Again, the instructor must be notified immediately. To insure your safety, neutralize acid or base spills before cleaning them up. Boric acid solution is available to neutralize base spills, and carbonate powder is provided to neutralize acids.



INSTRUCTIONS FOR RECORD WRITING

- 1. Write on the right hand page the following order:
- a) Serial number and date of performance (in the margin)
- b) Name and number of the experiment as given in the list.
- c) Aim of the experiment.
- d) Description of the apparatus.
- e) Principle of the experiment
- f) Procedure including sources of error and precautions taken to eliminate or to minimize them.
- g) Inference or Result.
- h) Explanation, if necessary of any divergence in the expected result.
- 2. Left hand page should contain the following in their proper places.
- a) Neat diagram of the main apparatus.
- b) Observation in tabular form.
- c) Calculation in tabular form.
- d) Graph sheets and other papers to be attached.

3. Students should submit a record of the previous experiments when they come for practical work.

4. An experiment is deemed to be complete when it is satisfactorily performed and recorded.

KEEP THE RECORD BOOK NEAT IT FETCHES MARKS

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INTRODUCTION TO CHEMISTRY LABORATORY

In all chemistry laboratories chemical analysis is carried out. Chemical analysis is the resolution of a chemical compound into its proximate or ultimate parts. It is divided into two types.

Qualitative Analysis: It deals with identification and conformation of the nature of a substance present in a given sample.

Quantitative Analysis: It deals with the determination of how much of each component is present in a given sample. Quantitative Analysis is further divided into two types.

Volumetric Analysis: It is based on measuring the volume of the solution of a substance.

Gravimetric Analysis: It is based on estimation of the amount of a given compound from the results of weighing.

Terms used in Volumetric Analysis

Titration: It is a process of adding one solution from the burette to another in the conical flask in order to complete the chemical reaction.

Titrant: The solution of known strength is called as titrant.

Titrate: The solution which contains the substance to be estimated is called as titrate.

Standard Solution: A solution whose concentration is known is called a standard solution. It is of two types

Primary standard solution: The substance whose standard solution can be prepared by direct weighing is known as Primary standard. They provide a reference to determine unknown concentrations or to calibrate analytical instruments and the composition of its solution should not change on standing or during storage. It is non-hygroscopic.

Examples: Oxalic acid, Potassium dichromate, Zinc sulfate, sodium carbonate etc.

Secondary Standards solution: A secondary standard is a standard, their solutions are not prepared directly by weighing and the exact strength is determined by titrating against a primary standard and the process is called standardization. It should change on standing or during storage. It is hygroscopic.

Example: NaOH, KOH, KMnO4, HCl, H₂SO₄ etc.

Indicator: Indicator is a substance which indicates the completion of reaction in a titration by color change.

Error: Error is not the same as a "mistake." It does not mean that you got the wrong answer. The error in measurement is a mathematical way to show the uncertainty in the measurement.

Percentage of Error: Error in measurement may also be expressed as a percent of error.

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% of Error = [Actual value – Measured value] X 100/ Actual value

Accuracy: Accuracy is how close a measured value is to the actual (true) value. The word accuracy means correctness. It means that there are no errors. The accuracy of a measurement system is the degree of closeness of measurements of a quantity to that quantity's actual (true) value.

Precision: Precision is how close the measured values are to each other. The precision of a measurement system, also called reproducibility or repeatability, is the degree to which repeated measurements under unchanged conditions show the same results. Precision measurements are those which are repeatable - so all measurements are clustered around the same value.

Concentration of a standard solution is generally expressed as:

Morality: It is the no of gram molecules of solute present in one liter of solution

Molarity M = <u>weight of the substance *1000</u>

Gram molecular weight *Volume in ml

Normality: It is defined as the no of gram equivalents of solute present in one liter of solution.

Normality N = weight of the substance X 1000

Gram equivalent weight X Volume in ml

Equivalent weight of an acid = molecular weight of an acid/ Number of replaceable H^+ ions Equivalent weight of a base = molecular weight of a base/ Number of replaceable OH^- ions Equivalent weight of an oxidizing or reducing agent = molecular weight of a substance/ No. of electrons gained or loosed

Types of Volumetric Titrations:

- Acid-Base Titrations
- Redox titrations
- Complexometric Titrations

Acid-Base Titrations: Titration is a process of neutralization. This method is used for determining an acid with alkali or base with an acid to produce salt and unionized water.

$NaOH + HCl \rightarrow NaCl + H_2O$

An indicator is (often) a weak acid or weak base that is placed into the unknown solution to determine the endpoint of the titration.

Phenolphthalein is a weak acid .it gives pink color in alkaline medium and colorless in acidic



medium and the Ph range is 8.0 - 9.6



Colorless in acidic medium (H⁺) attached

pink in basic media (H⁼) ion is removed



Structure of phenolphthalein indicator

Methyl orange indicator: It is a weak base. It gives yellow color in basic media and red color in acidic media and the pH range 3.1-4.4.



Red Color in acidic medium (H⁺) attach yellow

yellow color in basic media (H⁼) removed



Structure of Methyl orange indicator

Red-ox titration: It is also known as Oxidation – Reduction reaction. Titration a reducing agent (loosing of electrons) by an oxidizing agent (gaining of electrons) or titration of an oxidizing agent by an agent is known as Redox titration. The common of the redox titrations are

Permanganometry: $KMnO_4$ is the oxidizing agent and titrated against a reducing agent like Fe²⁺ (ferrous ammonium sulfate) etc.

Dichrometry: $K_2Cr_2O_7$ is the oxidizing agent and titrated against a reducing agent like Fe²⁺ (ferrous ammonium sulfate) etc.

Iodometry: It is based on oxidation by the action of free iodine generated from KI.

Type of Redox Indicators:

Self-Indicators: Many a times the titrant itself may be so strongly colored that after the equivalence point, a single drop of the titrant produces an intense color in the reaction mixture.

Ex: Potassium permanganate.



Internal indicator: Such indicators are added into the reaction mixtures Such indicators always have reduction potential values lower than the analyte system so that they react with the titrant only when whole of the analyte has been consumed, producing a readily detectable color change.

External indicator: In case a suitable redox indicator is not available for a given system, an indicator may be employed which will indicate the completion of reaction by physically or chemically reacting with the analyte (not through redox reaction). This reaction between indicator and the analyte may sometimes be an irreversible one and in some cases may even lead to precipitation. In those case indicators are not added to the reaction mixture on the whole, rather used externally on a grooved tile. Such indicators are called external indicators.

Complexometric Titrations:

The technique involves titrating metal ions with a complexing agent or chelating agent (Ligand) and is commonly referred to as complex metric titration. Ligand used widely in complexometric titrations is EDTA (ethylene diamine tetra acetic acid), because it forms stable complexes with a number of metal atoms at a definite pH range. The indicator used in this titration is EBT (eriochrome Black T)



Structure of EDTA

Structure of EBT

Applications of Complexometric Titrations:

- 1. Complexometric titrations have been employed with success for determination of various metals like Ca, Mg, Pb, Zn, Al, Fe, Mn, Cr etc.
- 2. Determination of total hardness of water by Complexometric method.



Apparatus:





EXPERIMENT-1

PREPARATION OF A POLYMER (BAKELITE)

Aim: To prepare the phenol – formaldehyde resin

Principle: Phenol formaldehyde resins (PF) include synthetic thermosetting resins such as Bakelite obtained by the reaction of phenols with formaldehyde. Phenol-formaldehyde resins are formed by a step-growth polymerization reaction that can be either acid- or base-catalysed.

Phenol is reactive towards formaldehyde at the ortho and para sites (sites 2, 4 and 6) allowing up to 3 units of formaldehyde to attach to the ring. The initial reaction in all cases involves the formation of ortho & para hydroxymethyl phenols. The ortho hydroxymethyl phenol undergoes linear polymerization to form novalac which is fusible and soluble in most of the organic solvents. Novolacs are phenol-formaldehyde resins made where the molar ratio of formaldehyde to phenol of less than one. Hexamethylene tetramine or "hexamine" is a hardener that is added to crosslink novolac. At ≥ 180 °C, the hexamine forms crosslink's to form methylene and dimethylene amino bridges. Base-catalysed phenol-formaldehyde resins are made with formaldehyde to phenol ratio of greater than one (usually around 1.5). These resins are called resols. When the molar ratio of formaldehyde: phenol reaches one, every phenol is linked together via methylene bridges, generating one single molecule, and the system is entirely cross linked. In Bakelite, this ratio is greater than one, and so it is very hard **Procedures**

Procedure:

- 5.0 ml of phenol is weighed and transferred into a clean and dried 250 ml beaker.
- 7 ml of formaldehyde solution is added carefully. (Caution : Avoid the inhaling the vapors and spilling of it on body)
- 5.0 ml of glacial acetic acid and one or two spatula of hexamine (hexamethylene tetramine) are added
- The contents of the beaker are heated gently in a water bath.
- The beakers are removed from the water bath and add conc. hydrochloric acid slowly drop-wise with constant stirring.
- After the addition of hydrochloric acid a white substance precipitates first. Finally, a pink colored plastic clump is formed at the bottom of the beaker while stirring.
- The plastic clump is now washed with distilled water for several times and then dried in a oven
- Now the sample is weighed and its weight is reported



Step-1: formation of o, p methyl phenol:



Step-2: formation of novalac:



Step-3: formation of Bakelite:



Precautions:

- Formaldehyde solution 37 % is very toxic by inhalation, ingestion and through skin absorption. Readily absorbed through skin. Probable human carcinogen. Mutagen. May cause damage to kidneys, allergic reactions, sensitization and heritable genetic damage.
- Phenol is acute poisoning by ingestion; inhalation or skin contact may lead to death. Phenol is readily absorbed through the skin. Highly toxic by inhalation.
- Safety glasses and protective gloves required. The experiment should be performed under a portable fume cupboard giving all-round visibility

S.	Given Amount	Reported Amount	% Error	Signature of the
No				Faculty



EXPERIMENT-2

DETERMINATION OF HARDNESS OF A GROUNDWATER SAMPLE

Aim: To determine the temporary and permanent hardness of given water sample.

Apparatus: Burette, pipette, burette stand, glazed tile, conical flask,

Chemicals: 0.1M Standard zinc sulfate solution, EDTA solution, Ammonia buffer, Erichrome Black T indicator.

Principle:

Metal ions form a complex with EDTA according to the equation $M^{2+} + EBT \rightarrow M$ -EBT complex Wine -red M-EBT complex + EDTA \rightarrow M-EDTA complex + EBT Stable complex Blue

The completion of the reaction between M^+ and EDTA is detected by the use of metal ion indicator namely Eriochrome Black T. Initially, when the P^H of the medium maintained at P^H =7to11 the metal ion combine with indicator to form metal indicator complex which appears as a wine red color. Near the end point, EDTA breaks the metal indicator complexation, resulting in the formation of metal-EDTA complex. Hence at the end point, the liberated free indicator yields a blue color to the solution. Thus the end point is a fine, sharp change from wine red to blue color.

Procedure:

Step 1: Standardize The EDTA Solution Using Standard Zinc Sulfate Solution.

Burette: The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: Conical flask is washed with distilled water and then pipette out 20ml of Zinc sulfate solution into the conical flask and then add 3.0 ml of ammonia buffer solution, 3-4 drops of Erichrome Black T indicator.

Indicator: Erichrome Black T indicator.

Endpoint: wine red to blue color.

To the conical flask containing Zinc sulfate, ammonia buffer solution, Erichrome Black T indicator is placed under the burette on a glazed tile, and then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from wine red to blue color which is the endpoint. The experiment is repeated until concurrent readings are obtained.



S NO	Volume of Zinc sulfate	Burette readings		Volume of EDTA solution
5.110	solution (V_1) in ml	Initial	Final	rundown (V ₂) in ml
1				
2				

Calculations:

The Molarity of EDTA solution can be calculated from the formula $V_1M_1 = V_2M_2$

Zinc sulfate

 V_1 =Volume of zinc sulfate solution = 20.0 ml M_1 =Molarity of zinc sulfate solution =0.1N

EDTA

 V_2 =Volume of EDTA solution = ml M_2 =Molarity of EDTA solution = M

Molarity of EDTA $M_2 = \frac{M_1 V_1}{V_2}$ = M

Step 2: Determination of the Total Hardness of Given Water Sample

Burette: The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: Conical flask is washed with distilled water and then pipette out 20ml of Zinc sulfate solution into the conical flask and then add 3.0 ml of ammonia buffer solution, 3-4 drops of Erichrome Black T indicator.

Indicator: Erichrome Black T indicator.

Endpoint: wine red to blue color.

To the conical flask containing water sample, ammonia buffer solution, Erichrome Black T indicator is placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from wine red to blue color which is the endpoint. The experiment is repeated until concurrent readings are obtained.

	Volume of Water	Burette readings		Volume of EDTA solution	
S. No	Sample (V_1 ml)	Initial	Final	rundown (V ₂) in ml	
1					
2					

Calculation:

The concentration of water solution can be calculated from the equation, $V_1 M_1 = V2 M_2$



Water sample:

 V_3 = Volume of Water sample = 20.0ml M_3 = Molarity of water sample = (?)

EDTA:

 V_2 = Volume of EDTA Solution =

M₂= Molarity of EDTA Solution =

Therefore $M_3 = \frac{V_2 M_2}{V_3} = M$

Hardness of water is expressed in terms of equivalent of calcium carbonate as ppm (parts per million)

Total Hardness of Water = $M_3 X 100 \text{ g/lit}$

= $M_3 X 100 X 1000 mg/lit$ or ppm

= ppm

Result:

Total hardness present in the given water sample = _____ ppm

S. No	Given Amount	Reported Amount	% Error	Signature of the Faculty



EXPERIMENT-3

DETERMINATION OF STRENGTH OF AN ACID IN Pb-ACID BATTERY

Aim: Determine the amount of H₂SO₄ present in lead-acid storage cell using 4N NaOH
Apparatus: Burette, pipette, burette stand, conical flask, wash bottle, glazed tile
Chemicals: H₂SO₄, 4N NaOH, distilled water, Phenolphthalein indicator.
Principle: H₂SO₄, reacts with Na₂CO₃ the following manner

 $H_2SO_4 + 2 \text{ NaOH} \rightarrow \text{Na}_2SO_4 + 2H_2O$

Procedure:

Step 1: Standardization of NaoH Using Standard 2N Oxalc Acid

Burette: The burette is cleaned first with distilled water and filled with NaOH, without any air bubbles.

Conical Flask: Conical flask is washed with distilled water and then pipette out 20 ml of oxalic acid solution into the conical flask and add 2 or 3 drops of Phenolphthalein indicator. Indicator: Phenolphthalein

End Point: Colourless to Pale pink

The conical flask is placed under the burette on a glazed tile, then the NaOH present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until a color changes from Colorless to Pale pink, which is the endpoint. The experiment is repeated till the concurrent readings are obtained.

~ ~ ~	Volume of Oxalic acid	Burette Readings		Volume of NaOH	
S. No	Solution in ml	Initial (a)	Final (b)	rundown in ml (b-a)	

Calculations:

Normality of NaOH is calculated using the equation

 $N_1V_1 = N_2V_2$

Oxalic acid:

Normality of Oxalic acid $N_1 = 2N$

Volume of Oxalic acid solution V_1 = 20 ml

NaOH:

Normality of NaOH $N_2 =$

Volume of NaOH V_2 = ml (burette readings)

=

Normality of NaOH (N₂) = N_1V_1/V_2

Ν



Step 2: Determination of H₂SO₄ Using Standard Solution of NaoH

Burette: The burette is cleaned first with distilled water and filled with NaOH, without any air bubbles.

Conical Flask: Conical flask is washed with distilled water and then pipette out 20ml of H_2SO_4 solution into the conical flask and add 2 or 3 drops of Phenolphthalein indicator.

Indicator: Phenolphthalein

End Point: Colourless to Pale pink

The conical flask is placed under the burette on a glazed tile, then the NaOH present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until a color changes from Colorless to Pale pink, which is the endpoint. The experiment is repeated till the concurrent readings are obtained.

S No	Volume of H ₂ SO ₄	Burette Readings		Volume of NaOH	
5.110	Solution in ml	Initial (a)	Final (b)	rundown in ml (b-a)	

Calculations:

Normality of H₂SO₄, is calculated using the equation

 $N_2V_2 = N_3V_3$

NaoH

 N_2 =Normality of NaOH = N

 V_2 =Volume of NaOH solution = ml

$H_2SO_4\\$

 N_3 =Normality of H_2SO_4 =

 $V_3 = Volume of H_2SO_4 = ml$

Normality of H_2SO_4 (N₃) = N₂V₂/V₃

=

S. No	Given Amount	Reported Amount	% Error	Signature of the
				Faculty



EXPERIMENT-4

DETERMINATION OF ACID NUMBER OF LUBRICATING OIL

Aim: To determine the Acid number of lubricating oil.

Apparatus: 50 ml burette, 20ml pipette, 250 ml conical flask, 100ml beaker, 250 ml beaker, 50 ml beaker and 50 ml measuring jar.

Chemicals: KOH solution, 0.02N oxalic acid, Oil sample, Phenolphthalein indicator, Ethyl alcohol.

Principle:

The Acid number of lubricating oil is defined as the number of milligrams of potassium hydroxide required to neutralize the free acid present in 1 g of the oil sample. In good lubricating oils, the acid number should be minimum (<0.1). Increase in acid value should be taken as an indicator of oxidation of the oil which may lead to gum and sludge formation besides corrosion. Since free fatty acids present in the oil react with base, their quantity can be estimated by titrating the known weight of the oil sample dissolved in a suitable solvent with a standard alcoholic solution of KOH to a definite end point

$$RCOOH + KOH \rightarrow RCOOK + H_2O$$

Procedure:

Step 1: Standardization of KOH

- 20 ml standard oxalic acid solution is pipette out into a 250 ml of conical flask and few drops of Phenolphthalein indicator is added.
- The above solution is titrated with standard KOH solution taken in the burette until the solution changes from colorless to light pink colour
- The same procedure is repeated until any two readings coincide
- The concentration of KOH is calculated.

Observation and Calculation:

Step-1: Standardization of KOH Solution.

- Burette : KOH solution
- Conical flask : 20 ml. of Oxalic Acid
- Indicator : Phenolphthalein
- End point : Colourless to pink.

C N		Burette	readings	Vol. of KOH
S. No.	Vol. of Oxalic Acid	Initial	Final	rundown



Normality of KOH N₂ = $\frac{N_1 \times V_1}{V_2}$

- N_1 = Normality of oxalic acid
- V_1 = Volume of the oxalic acid
- V_2 = Volume of the KOH

 $N_2 = N$

Step 2: Determination of Acid Number of given oil sample

- 1 gram (1.1 ml) of oil sample is taken in a 250 ml conical flask and dissolved in 5 ml of Ethyl alcohol.
- One or two drops of Phenolphthalein indicator is added and the solution is titrated with KOH taken in the burette until the solution changes from colorless to light pink
- The same procedure is repeated until any two readings coincide
- The Acid Number of oil sample is calculated.

Step-2: Determination of Acid Number:

Burette	:	Std. KOH solution
Conical flask	:	1gm. of lubricating oil + 5 ml of alcohol.
Indicator	:	Phenolphthalein
End point	:	Colourless to pink.

S. No	Vol. of	Burette readings		Vol. of VOU
5. INO.	lubricating oil	Initial	Final	

Acid Number of given oil sample is

(mg of KOH required to neutralize the acid present in 1 gm of oil) =

$$N_2 \times Eq. wt of KOH \times Vol. of KOH \times 100$$

1	000
T	000

	=	
Vol. of KOH	=	titer value in the above titration
Eq. wt of KOH	=	56.01
N_2	=	Normality of KOH

Result:

Name of lubricating oil sample	Weight of oil sample	Acid number	Marks awarded	Signature of the faculty



EXPERIMENT-5

DETERMINATION OF VISCOSITY OF LUBRICATING OIL BY REDWOOD VISCOMETER 1/ REDWOOD VISCOMETER 2

Aim: To determine the kinematic viscosity of given lubricating oil at a given temperature by using Redwood Viscometer

Principle: The internal drag arises between two successive layers of the liquid is known as viscosity. Further, the force per unit area required to maintain the velocity gradient by one unit between two successive layers of one unit length apart is known as viscosity coefficient. High viscous liquids move slowly while low viscous liquids move fast through a given capillary. Further, the time required to flow a given volume of liquid through a capillary depends on its viscosity. Therefore, the viscosity of liquid can be determined by determining the time required to flow the known volume of liquid through a standard capillary. Viscosity is expressed in poise.

Procedure:

- The Redwood viscometer consists of oil cup which is opened at the upper end and it is fitted with an orifice
- It is cleaned thoroughly with suitable solvent and then dried
- The orifice is covered with brass ball to stop the flow of oil
- The oil cup is placed in the cylindrical copper vessel which serves as water bath
- The bath is filled with suitable liquid which has the boiling point higher than the temperature at which the viscosity of oil to be determined
- If the viscosity of the oil is to be determined at 80[°]C or below, the bath is filled with water
- The instrument level is adjusted on the tripod stand with the help of the leveling screws
- Now the oil cup is filled with oil to be tested carefully up to the level indicated and the covered with lid
- Two thermometers, one is in the oil and the other one is in the liquid (water) are immersed
- Similarly two stirrers also placed in the oil and the liquid
- One 50 ml flask is kept in position below the jet
- Now the oil is heated slowly with constant stirring of oil and the water until it reaches to the required temperature at which the viscosity of the oil is to be determined
- When the temperature of the oil has quite steady and reaches the required temperature, the brass ball is lifted and simultaneously the stop watch is started.



- The oil is allowed to flow through the orifice and collected in the flask
- Stop watch is stopped when 50 ml of oil is collected in the flask up to the mark and immediately the orifice is covered with brass ball to stop the over flow of the oil
- The time required to flow the 50 ml of oil is noted
- The oil cup is refilled again with oil and same procedure is repeated for five to six times

The viscosity of oil is calculated at given temperature

Observation and Calculations:

S. No.	Temperature	Time required to flow 50 ml of oil (in Seconds)	Kinematic Viscosity (Centistokes) V = At – B/t	Average Kinematic Viscosity (Centistokes)

The kinematic viscosity of the liquid is given by the formula

$$V = At - B/t$$

V = Kinematic viscosity of oil in centistokes

t = Time of flow for 50 ml of oil in seconds

A and B are instrument constants

S. No	Type of equipment	Time of flow	A value	B value
1	Redwood 1	40 to 85 secs	0.264	190
2	Redwood 1	85 to 2000 secs	0.247	65
3	Redwood 2		0.027	20

Precautions

- The oil should be filtered through a 100 mesh wire sieve before testing for its viscosity
- Receiving flask should be placed in such a way that the oil jet touches inside layer of the flask and does not form foaming
- Same receiving flask should be used for all readings



• After each reading, oil should be completely drained out of the receiving flask and it should be thoroughly cleaned and dried

Name of the	Average of kinematic	Marks awarded	Signature of the
Lubricating Oil	viscosity (Centistokes)		faculty



EXPERIMENT-6

DETERMINATION OF FLASH AND FIRE POINTS OF LUBRICATING OIL BY CLEVELANDS APPARATUS

Aim: To find the flash point and fire point of the given oil sample by using Cleveland`s apparatus

Definition: Fire Point is the lowest temperature at which application of test film causes the material to ignite and burn at least for 5 secs under specified conditions to the test.

Apparatus: Cleveland's apparatus, thermometer, Oil sample (petrol, diesel and kerosene etc) **Procedure:** The oil cup is filled with sample, so that the meniscus is exactly at the filling line at room temperature. Care is taken that no sample is above the filling line or on the outside of the apparatus. The sample is heated by adjusting the energy regulator so that the raise in temperature does not exceed 17°C/min till the temperature reaches approximately 37.7°C less than the flash point of the sample. There after the rate of the heating is decreasing for at least the last 28°C below the flash point is reached it shall not be less than 5°C/min. However if the flash point of the given oils are lower than 65°C, the rate of heating should give 2°C/min rise in the beginning and 0.5°C/min in final stage. The test flame is applied and the flash point is obtained. After determine the flash point heating is continued so that the rise in temperature is maintained at the specific rate and rise point is obtained .The experiment with oil is continue until the successive minimum temperatures are equal.

Observation Table

Determination of Flash point and Fire point

S. No	Temperature in Degree Celsius	Inference



Result: The fire and flash point of the given oil sample

S. No	Sample used	Flash Point	Fire point

Precautions:

- 1. As moisture affects the flash point, all the parts of the cup and its accessories should be dried before placing oil in the cup.
- 2. Always a fresh portion of the oil sample should be used.
- 3. A second determination on the same portion of oil shows a higher flash point.
- 4. The thermometer bulb should dip into the oil.
- 5. For applying the test flame, the slide should be drawn open slowly and closed quickly.
- 6. Stirring should be discontinuing during the application of the test flame.



EXPERIMENT-7

DETERMINATION OF THE AMOUNT OF ACIDITY OF A GIVEN WATER SAMPLE

Aim : To determine the acidity of the given water sample.

Apparatus : Burette, Pipette, Conical flask, Beakers, Wash bottle, Burette stand.

Chemicals : Standard sodium hydroxide (0.02 N), Phenolphthalein indicator, Methyl orange indicator, sodium thiosulphate (0.1 N), water sample.

Principle : The acidity of a solution is a measure of its capacity to neutralize bases.

Acidity is due to the presence of mineral acids like H_2SO_4 , HCI, HNO_3 and dissolved CO_2 in the form of H_2CO_3 . These acids can be estimated by titration against standard sodium hydroxide using methyl orange and phenolphthalein indicators.

Titration to P^{H} 4.5 or a sharp change from faint orange to yellow of methyl orange indicator will indicate the neutralization of mineral acids.

 $H^+ + OH^- \xrightarrow{p^H 4.5} H_2O$

..... (1)

Titration to P^H 8.5 or the appearance of faint pink colour, by the use of phenolphthalein will indicate total acidity. (Complete neutralization of mineral acids and dissolved CO_2)

Interference due to presence of residual Chlorine can be removed by the addition of one drop of Sodium thiosulphate (Hypo) solution to the water sample.

The acidity is measured interms of CaCO3 equivalents and expressed in ppm or mg/litre.

Formula:

Acidity of water sample interms of CaCO3 equivalents

Titre value × conc. of NaOH × 50 × 1000 volume of sample ppm

where 50 is the equivalent weight of CaCO3.

Procedure :

Part A: Methyl Orange Acidity:

- 1. Rinse and fill the burette with 0.02 N NaOH solution.
- 2. Pipette out 20 ml of water sample into a clean conical flask.
- 3. Add a drop of hypo solution and 2 drops of methyl orange indicator. The solution colour changes to orange.



- 4. Titrate this solution against 0.02 N NaOH solution till th colour changes from faint orange to yellow indicating P^{H} of 4.5.
- 5. Note the volume of NaOH consumed (V_1 ml).
- 6. Repeat the titration until to obtain concordant readings.
- 7. Calculate methyl orange acidity by using the formula given below :

Methyl orange acidity =
$$\frac{V_1 \times 0.02 \times 50 \times 1000}{20} ppm.$$

Observations & Calculations :

Part A: Methyl orange acidity (Mineral acidity)

Burette = 0.02 N NaOH solution

Conical flask = 20 ml water sample + Drop of $Na_2S_2O_3$

Indicator = Methyl orange

End point = Faint orange to yellow

S.No.	Volume of Water Sample	Burette readings (ml)		Volume of NaOH	
(ml)	Initial	Final	V ₁ ml		
1.	and the production of the second second	The parallel		and the second	
2.					
3.	2				
4.				No. +	

Methyl orange acidity = $\frac{Titre value \times conc. of NaOH \times 50 \times 1000}{1000}$



 $= \frac{V_1 \times 0.02 \times 50 \times 1000}{20} = _ppm \text{ (or) } mg/litre.$

Part B: Phenolphthalein Acidity (Total Acidity):

- 1. Rinse and fill the burette with 0.02 N NaOH solution.
- 2. Pipette out 20 ml of water sample into a clean conical flask.
- 3. Add a drop of hypo solution and a drop of phenolphthalein indicator.
- 4. Titrate this solution against 0.02 N NaOH solution till the appearance of faint pink colour indicating P^{H} of 8.5.
- 5. Note the volume of NaOH consumed (V_2 ml).
- 6. Repeat the titration to obtain concordant readings.
- 7. Calculate total acidity by using the formula given below :

Total acidity = $\frac{V_2 \times 0.02 \times 50 \times 1000}{20}$ ppm.



	Conical flask = 20 ml wate Indicator = PhenoIph End point = Colourles	er sample + Dro thalein s to pink	op of Na ₂ S ₂ O ₃	
S.No.	Volume of Water Sample (ml)	Burette rea Initial	dings (<i>ml</i>) Final	Volume of NaOH V2 ml
1.			and the same	
2.		and the second	Mar Maria	
3.			,	



$$=\frac{v_1 \times 0.02 \times 50 \times 1000}{20} = _____ppm$$
 (or) mg/litre.

Acidity due to carbonic acid = Total acidity - Mineral acidity

_____ ppm (or) mg/litre.

Result:

- 1. Methyl orange acidity or Mineral acidity = ____ ppm.
- 2. Phenolphthalein acidity or Total acidity = _ _ _ ppm.
- 3. Carbonic acid acidity of water sample = ____ ppm.

S. No	Given Amount	Reported Amount	% Error	Signature of the Faculty



EXPERIMENT-8 DETERMINATION OF THE AMOUNT OF ALKALINITY OF A GIVEN WATER SAMPLE

Aim : To determine the alkalinity of the given water sample.

Apparatus : Burette, Pipette, Conical flask, Glazed tile, Beakers, Wash bottle, Burette stand.

Chemicals : Standard sulphuric acid (0.02 N), Phenolphthalein and Methyl orange indicators, sample water, distilled water.

Principle : The alkalinity of a solution is a measure of its capacity to neutralize acids. The alkalinity is due to OH^- , CO_3^{2-} , HCO_3^- ions. It is determined by titration against standard sulphuric acid using phenolphthalein and methyl orange indicators.

Titration to P^H 8.3 with phenolphthalein or the disappearance of pink colour will indicate phenolphthalein alkalinity.

$$OH^{-} + H^{+} \xrightarrow{p^{H} 8.3} H_{2}O \qquad \dots \dots \dots (1)$$

$$CO_{3}^{2^{-}} + H^{+} \xrightarrow{p^{H} 8.3} HCO_{3}^{-} \qquad \dots \dots \dots (2)$$

Titration to P^H 4.5 with methyl orange or the appearance of pink colour will indicate total alkalinity (i.e., complete neutralization of OH^- , CO_3^{2-} and HCO_3^- ions).

$$HCO_3^- + H^+ \xrightarrow{p^{n-4.5}} H_2CO_3 \rightarrow H_2O + CO_2 \uparrow$$
(3)

Formula:

The amount of alkalinity interms of CaCO3 equivalents

 $\frac{Titre \ value \times conc. \ of \ H_2SO_4 \times 50 \times 1000}{volume \ of \ sample} \ ppm \,.$

Procedure :

Part A - Phenolphthalein Alkalinity:

- 1. Rinse and fill the burette with $0.02 N H_2SO_4$ solution.
- 2. Pipette out 20 ml of water sample into a clean conical flask.
- 3. Add a drop of phenolphthalein indicator to the sample.
- If pink colour develops titrate against 0.02 N H₂SO₄ till the pink colour disappears indicating P^H 8.3.
- 5. Note the volume of H_2SO_4 used (V_1 ml).
- 6. Repeat the titration to obtain concordant readings.
- 7. Calculate phenolphthalein alkalinity by using the formula given below :

Phenolphthalein alkalinity = $\frac{V_1 \times 0.02 \times 50 \times 1000}{20}$ ppm.



Observations & Calculations :

Part A - Phenolphthalein Alkalinity:

Burette = $0.02 N H_2 SO_4$ solution

Conical flask = 20 ml of sample

Indicator = Drop of phenolphthalein

End point = Pink to colourless

• S.No.	Volume of Sample	Burette readings (ml)		Volume of H ₂ SO ₄ Used	
	(111)	Initial	Final	(V ₁ <i>inl</i>)	
.1.					
2.					
3.			Prix a	Day & the Instally and	
4.					

0.02 × 50 × 1000

Phenolphthalein alkalinity interms of

$$CaCO_3$$
 equivalents = $\frac{V_1 \times 0.02 \times 50 \times 1000}{2}$ $=$ $\frac{7.5}{2}$

Phenolphthalein alkalinity = ____ ppm.

Part B - Total Alkalinity:

- 1. Rinse and fill the burette with $0.02 N H_2SO_4$ solution.
- 2. Pipette out 20 ml sample into a clean conical flask.
- 3. Add 2 to 3 drops of methyl orange indicator to the sample.
- 4. If yellow colour develops, titrate against 0.02 N H_2SO_4 till the pink colour appears indicating P^H of 4.5.

1.

- 5. Note the volume of H_2SO_4 used (V_2 ml).
- 6. Repeat the titration to obtain concordant readings.
- 7. Calculate total alkalinity by using the formula given below :

Total alkalinity =
$$\frac{V_2 \times 0.02 \times 50 \times 1000}{20} ppn$$

Part B - Total Alkalinity :

Burette = $0.02 N H_2 SO_4$ solution

Conical flask = 20 ml of sample

Indicator = 2 to 3 drops of Methyl orange

End point = Yellow to pink



S.No.	Volume of Sample	Burette rea	dings (ml)	Volume of H ₂ SO ₄ Used	
	(<i>ml</i>)	Initial	Final	V ₂ ml	
1.		9	Trail and		
2.			1-2010	1.	
3.	- Contraction	and			
4.			Part and		
	Total alkalinity interms of				
	$CaCO_3$ equivalents = $\frac{V_2 \times 0}{V_2}$	0.02×50×1000			
	=	 ppm		20	
.:	Total alkalinity = pp	m.		a free states of a	

Result:

Phenolphthalein alkalinity of water sample in terms of CaCO3 equivalents = _____ ppm

Total alkalinity of water sample in terms of CaCO3 equivalents = _____ ppm

S. No	Given Amount	Reported Amount	% Error	Signature of the Faculty



EXPERIMENT-9 DETERMINATION OF AMOUNT OF COPPER IN A GIVEN SAMPLE

Aim: To Determine the amount of copper present in a sample by using EDTA

Apparatus: Burette, pipette, burette stand, glazed tile, conical flask,

Chemicals: 0.1M zinc sulfate solution, EDTA solution, Ammonia buffer, Erichrome Black T

indicator, Copper ore, distilled water, fast sulfan black F indicator

Principle: Metal ions form a complex with EDTA according to the equation

 M^{2+} + EBT \rightarrow M-EBT complex (Wine –red)

M-EBT complex + EDTA \rightarrow M-EDTA complex + EBT

Stable complex Blue

The completion of the reaction between M^+ and EDTA is detected by the use of metal ion indicator namely Eriochrome Black T. Initially, when the P^H of the medium maintained at P^H =7to11 the metal ion combine with indicator to form metal indicator complex which appears as a wine red color. Near the end point, EDTA breaks the metal indicator complexation, resulting in the formation of metal-EDTA complex. Hence at the end point, the liberated free indicator yields a blue color to the solution. Thus the end point is a fine, sharp change from wine red to blue color.

Procedure:

Step 1: Standardize the EDTA Solution Using Standard Zinc sulfate Solution

Burette: The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: Conical flask is washed with distilled water and then pipette out 20 ml of Zinc sulfate solution into the conical flask and then add 3.0 ml of ammonia buffer solution, 3-4 drops of Erichrome Black T indicator.

Indicator: Erichrome Black T indicator.

Endpoint: wine red to blue color.

To the conical flask containing Zinc sulfate, ammonia buffer solution, Erichrome Black T indicator is placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from wine red to blue color which is the endpoint. The experiment is repeated until concurrent readings are obtained.



	Volume of Zinc sulfate	Burett	e readings	Volume of EDTA
S. No	solution (V_1) in ml	Initial	Final	solution rundown (V ₂) in ml

Calculations:

The Molarity of EDTA solution can be calculated from the formula $M_1V_1 = M_2V_2$

Zinc sulfate:

Volume of zinc sulfate solution $V_1 = 20.0$ ml

Molarity of zinc sulfate solution $M_1 =$

EDTA:

Volume of EDTA solution $V_2 =$

Molarity of EDTA solution $M_2 =$

Molarity of EDTA $M_2 = M_1 V_1$

$$V_2$$

= M

Step 2: Determination of the Amount of Copper Present in a Given Copper Sample Using Fast Sulfan Black -F Indicator

The copper ion solution given in the 100ml volumetric flask is dilute up to the mark and mix well to make the uniform solution

Burette: The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: onical flask is washed with distilled water and then pipette out 20ml of copper ion solution into the conical flask, add 20ml of distilled water, 5ml of concentrated ammonia solution and add 5-6 drops of the indicator.

Indicator: fast sulfan black -F

Endpoint: blue to a dark green

To the conical flask containing copper ion solution, distilled water, ammonia solution, fast sulfan black -F indicator is placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction



continuously, the titration is continued until the color changes from blue to a dark green color.

	Volume of Copper	Burette	e Readings	Volume of EDTA
S. No	sulfate solution (V_3) in	T., 141 - 1	\mathbf{E}^{t} and \mathbf{I}	solution rundown (V_2) in
	ml	Initial	Final	ml
	1111			1111

Calculations:

The copper ion can be determined by using the following formula: $M_2V_2 = M3V3$

Copper sulfate:

 V_3 =Volume of copper sulfate solution = 20 ml

M₃=Molarity of copper sulfate solution =?

EDTA:

 V_2 =Volume of EDTA solution = ml

 M_2 = Molarity of EDTA solution = M

The Molarity of copper sulfate solution, $M_3 = \underline{M}_2 \underline{V}_2$

V_3

= M

The amount of copper ion present in a given unknown solution =

$$M_3$$
 X gram atomic weight of Cu⁺² (63.54) X 100 = ____ gm

1000

Result: The amount of copper ion present in given 100ml solution = _____ gm

S. No	Given Amount	Reported Amount	% Error	Signature of the Faculty



EXPERIMENT-10 DETERMINATION OF AMOUNT OF ZINC IN A GIVEN SAMPLE

Aim: To Determine the amount of zinc using standard EDTA.

Apparatus: Burette, pipette, burette stand, glazed tile, conical flask,

Chemicals: 0.1M Standard zinc sulfate solution, EDTA solution, Ammonia buffer, Erichrome Black T indicator.

Principle: Metal ions form a complex with EDTA according to the equation

 $M^{2+} + EBT \rightarrow M-EBT$ complex

Wine -red

M-EBT complex + EDTA \rightarrow M-EDTA complex + EBT

Stable complex Blue

The completion of the reaction between M^+ and EDTA is detected by the use of metal ion indicator namely Eriochrome Black T. Initially, when the P^H of the medium maintained at P^H =7to11 the metal ion combine with indicator to form metal indicator complex which appears as a wine red color. Near the end point, EDTA breaks the metal indicator complexation, resulting in the formation of metal-EDTA complex. Hence at the end point, the liberated free indicator yields a blue color to the solution. Thus the end point is a fine, sharp change from wine red to blue color.

Procedure:-

Step 1: Standardize the EDTA Solution Using Standard Zinc sulfate Solution

Burette: The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: Conical flask is washed with distilled water and then pipette out 20ml of Zinc sulfate solution into the conical flask and then add 3.0 ml of ammonia buffer solution, 3-4 drops of Erichrome Black T indicator.

Indicator: Erichrome Black T indicator.

Endpoint: wine red to blue color.

To the conical flask containing Zinc sulfate, ammonia buffer solution, Erichrome Black T indicator is placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from wine red to blue color which is the endpoint. The experiment is repeated until concurrent readings are obtained.



	Volume of Zinc sulfate	Burette readings		Volume of EDTA
S. No	solution (V_1) in ml	Initial	Final	solution rundown (V ₂) in ml

Calculations:

The Molarity of EDTA solution can be calculated from the formula $V_1M_1 = V_2M_2$

Zinc sulfate

```
V_1=Volume of zinc sulfate solution = 20.0 ml
```

M1=Molarity of zinc sulfate solution= 0.1M

EDTA

 V_2 =Volume of EDTA solution = ml

 M_2 =Molarity of EDTA solution = ?

Molarity of EDTA $M_2 = M_1 V_1$

V₂ = = M

Step 2: Determination of Zinc present in the solution

The zinc solution given in the 100ml volumetric flask is dilute up to the mark and mix well to make the uniform solution

Burette: The burette is washed with distilled water, and then fills it with EDTA without air bubbles.

Conical Flask: Conical flask is washed with distilled water and then pipette out 20ml of Zinc sulfate solution into the conical flask and then add 3.0 ml of ammonia buffer solution, 3-4 drops of Erichrome Black T indicator.

Indicator: Erichrome Black T indicator.

Endpoint: wine red to blue color.

To the conical flask containing Zinc sulfate, ammonia buffer solution, Erichrome Black T indicator is placed under the burette on a glazed tile, then the EDTA present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until the color changes from wine red to blue color which is the endpoint. The experiment is repeated until concurrent readings are obtained.



	Volume of Zinc sulfate	Burett	e readings	Volume of EDTA
S. No	solution (V_3) in ml	Initial	Final	solution rundown (V ₄) in
		Introd	1 11141	ml

Calculations:

The Molarity of EDTA solution can be calculated from the formula $V_3M_3 = V_4M_4$

Zinc sulfate:

```
V_3 =Volume of zinc sulfate solution = 20.0 ml
```

 M_3 =Molarity of zinc sulfate solution = 0.1M

EDTA:

 V_4 =Volume of EDTA solution =

 M_4 =Molarity of EDTA solution =

Molarity of EDTA $M_3 = M_4 V_4$

$$V_3$$

=

Amount of zinc ion present in 100 ml of the given unknown solution = $M_3 X 65.38 X 100 g$

1000

Result:

The amount of zinc ion present in given 100ml solution = gm.

S. No	Given Amount	Reported Amount	% Error	Signature of the Faculty



EXPERIMENT-11

ESTIMATION OF FERROUS IRON BY DICHROMETRY

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

Apparatus: Beaker, burette, pipette, conical flask, volumetric flask.

Chemicals: K₂Cr₂O₇, diphenylamine, Conc.H2SO4, Distilled water.

Principle: Ferrous iron is oxidized to ferric iron by potassium dichromate in 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. The equivalent weight of iron is its atomic weight i.e.55.86 since one equivalent of potassium dichromate oxidizes one equivalent of iron.

Procedure:

- 1. Preparation of standard potassium dichromate: weigh out accurately about 0.49gms of potassium dichromate into a standard flask into a100 ml standard flask and dissolve the solid in a small quantity of distilled water make up the resulting solution with distilled water up to the mark and shake the flask well for uniform concentration.
- 2. Preparation of acid mixture: Mix up 100 ml of phosphoric with 300ml of concentrated H_2SO_4 in a reagent bottle and stopper it.
- 3. Preparation of diphenylamine: Dissolve 1gm of diphenylamine in 100ml of concentrated H₂SO₄

Step 1: Determination of Unknown Ferrous Iron in a Sample Using Std. K₂Cr₂O₇ solution

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

S No	Volume of Fe(II) solution Burette re		e readings	Volume of K ₂ Cr ₂ O ₇
5. NO	in ml	Initial	Final	solution rundown in ml

Calculations and Observations



1000

Calculations:

The Normality of Fe(II) sulfate solution can be calculated from the formula

 $\mathbf{V}_1\mathbf{M}_1 = \mathbf{V}_2\mathbf{M}_2$

K₂**Cr**₂**O**₇:

 V_1 =Volume of $K_2Cr_2O_7$ solution = 20.0 ml

 N_1 =Normality of $K_2Cr_2O_7$ solution = 0.1M

Fe(II) sulfate:

 V_2 =Volume of Fe(II) sulfate solution =

 N_2 =Normality of Fe(II) sulfate solution =

=

Normality of Fe(II) sulfate $N_2 = \underline{N_1}\underline{V_1}$

 V_2

Amount of Ferrous iron present in 100 ml of the given unknown solution = $N_2 X 65.38 X 100 g$

Result:

The amount of Ferrous iron present in given 100ml solution = _____ gm

S. No	Given Amount	Reported Amount	% Error	Signature of the Faculty



EXPERIMENT-12 DETERMINATION OF KMNO₄ BY USING STANDARD OXALIC ACID SOLUTION

Aim: To determine the concentration/molarity of KMnO4 solution by titrating it against a 0.1 M standard solution of oxalic acid.

Apparatus: Beaker, burette, pipette, conical flask, volumetric flask.

Chemicals: O.1 M Oxalic acid, O.1M KMnO₄, 1.0 M H2SO4, Distilled water.

Principle: In the present experiment, potassium permanganate acts as a powerful oxidizing agent. Although KMnO4 acts as an oxidizing agent in alkaline medium also, for quantitative analysis mostly acidic medium is used. The oxidizing action of KMnO4 in the acidic medium can be represented by the following equation:

 $MnO_4 + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$

The acid used in this titration is dilute sulfuric acid. Nitric acid is not used as it is itself an oxidizing agent and hydrochloric acid is usually avoided because it reacts with KMnO4 according to the equation given below to produce chlorine and chlorine which is also an oxidizing agent in the aqueous solution.

 $2KMnO4 + 16 \text{ HCl} \rightarrow 2KCl + 2 \text{ MnCl}_2 + 5Cl_2 + 8 \text{ H}_2O$

Since, oxalic acid acts as a reducing agent; it can be titrated against potassium permanganate in the acidic medium according to the following equation:

Reactions of oxalic acid

A. Chemical equations

Reduction half reaction: $2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5$ [O] Oxidation half reaction: $H_2C_2O_4$ + [O] $\rightarrow 2CO_2$ + $5H_2O$

 $2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2$

B. Ionic equation

Reduction half reaction: $MnO_4^- + 5e^- + 8H^+ \rightarrow Mn^{2+} + 4[H_2O] \times 2$ Oxidation half reaction: $C_2O_4^- \rightarrow 2CO_2 + 2e^- \times 5$

$$2 \text{ MnO}_4^- + 5\text{C}_2\text{O}_4^{2-} + 16\text{H}^+ \rightarrow 2\text{Mn}^{2+} + 10\text{CO}_2 + 8\text{H}_2\text{O}$$

In these equations, MnO_4^- is reduced to Mn^{2+} and $C_2O_4^{2-}$ is oxidized to CO_2 . The oxidation number of carbon in $C_2O_4^{2-}$ changes from +3 to +4. In these titrations, potassium permanganate acts as a self indicator. Initially color of potassium permanganate is discharged



due to its reduction by oxalic acid. After complete consumption of oxalate ions, the end point is indicated by the appearance of a light pink color produced by the addition of a little excess of un-reacted potassium permanganate. Further, during the titration of oxalic acid against potassium permanganate, warming of oxalic acid solution $(50^\circ-60^\circ\text{C})$ along with dilute H₂SO₄ is required. This is essential because the reaction takes place at higher temperature. During the titration, first manganese sulfate is formed which acts as a catalyst for the reduction of KMnO₄ by oxalic acid. Therefore, in the beginning the reaction rate is slow and as the reaction proceeds, the rate of the reaction increases.

Procedure:

Preparation of 0.1 M standard solution of oxalic acid:

- To determine the strength of potassium permanganate by titrating it against the standard solution of 0.1 M oxalic acid." we should calculate for 0.1 M of oxalic acid.
- The molecular mass of oxalic acid =126 g
- In order to prepare M/10 oxalic acid solution, 12.6 g of oxalic acid should be dissolved per litre of solution.

Titration of oxalic acid solution against potassium permanganate solution

- Rinse and fill a clean burette with potassium permanganate solution. Remove the air bubble, if any, from the nozzle of the burette by releasing some solution through it.
- Take 10 mL of 0.1 M oxalic acid solution in a conical flask and add 5 mL of 1.0 M H_2 SO₄ to it to prevent the formation of any precipitate of manganese dioxide during the course of the titration.
- Heat the oxalic acid solution up to 50°- 60°C before titrating it with potassium permanganate solution taken in the burette. To increase the visibility of the color change, place the conical flask containing the solution to be titrated over a white glazed tile kept below the nozzle of the vertically fitted burette.
- Note the initial reading of the volume of permanganate solution in the burette and add it in small volumes to the hot oxalic acid solution while swirling the contents of the flask gently. The violet color of permanganate solution is discharged on reaction with oxalic acid. The end point is indicated by the appearance of permanent light pink color due to a slight excess of permanganate solution.
- Repeat the titration till three concordant readings are obtained. Since the solution of KMnO4 is of dark color, the upper meniscus should be considered for noting the burette readings.



Calculations and Observations

S No	Volume of Oxalic acid	Burette readings		Volume of KMnO ₄
5.110	solution in ml	Initial	Final	solution rundown in ml

Calculations:

Oxalic acid

V₁=Volume of Oxalic acid solution =

M₁=Molarity of Oxalic acid EDTA solution =

 n_1 =Number of Moles of oxalic acid = 2

KMnO₄

 V_2 =Volume of KMnO₄ solution =

M₂=Molarity of KMnO₄ solution =

 n_2 =Number of Moles of KMnO₄ = 5

Molarity of KMnO₄ M₂ = $\underline{M_1V_1 * n2}$ V₃* n₁

Result:

(i) Molarity of KMnO₄ solution is _____

(ii) Amount of KMnO₄ in the solution is _____

S. No	Given Amount	Reported Amount	% Error	Signature of the Faculty



ESTIMATION OF CALCIUM IN PORT LAND CEMENT

Aim: To estimate calcium oxide in the given sample of cement solution.

Preparation of cement solution Cement solution is prepared by weighing accurate sample of cement, dissolved in minimum concentrated HCl, warmed and then diluted with distilled water.

STEP-1: Preparation of standard ZnSO4 solution:

Around 1.45g of Zinc sulphate hepta hydrate (ZnSO4. 7H2O) is weighed accurately and transferred into a 100 ml volumetric flask using a funnel. This is dissolved in a minimum amount of distilled water and finally made up to the mark.

Preparation of EDTA solution: Around 4.65g of EDTA disodium salt is weighed accurately and transferred into 250 ml volumetric flask using a funnel. This is dissolved in a minimum amount of distilled water and finally made up to the mark.

Preparation of Buffer solution: 17.42g of NH4Cl is weighed accurately and transferred into a beaker. To this 142 ml of NH4OH is added, mixed properly and is finally made up to 250 ml with distilled water. The pH is adjusted to 10.2 by adding either NH4Cl of NH4OH using a pH meter.

Preparation of 5M Sodium Hydroxide solution: 50g of NaOH is weighed and transferred into a 250mL beaker. This is dissolved in minimum distilled water and finally made up to the mark with distilled water.

Preparation of test sample solution: 1.25g of test cement sample is weighed accurately and transferred into a 500 ml beaker. This is dissolved in 20 ml of concentrated hydrochloric acid and then diluted up to 500 ml with distilled water

STEP-2: Standardization of EDTA solution:

10 ml of standard ZnSO4 solution is pipetted out into a 250 ml conical flask. To this 10 ml of distilled water is added. 5 ml of pH 10.2 buffer and 2–3 drops of EBT indicator is added and then the reaction mixture is titrated with EDTA from the burette until the wine red colored solution changes to blue. The volume of EDTA rundown is recorded and the titration is repeated until concurrent values are obtained and the values are tabulated.

S No	Volume of ZnSO ₄	Burette readings		Volume of EDTA
5.10	solution in ml	Initial	Final	solution rundown in ml



Date:

Calculations:

The Molarity of EDTA solution can be calculated from the formula $V_1M_1 = V_2M_2$

Zinc sulfate

 V_1 =Volume of zinc sulfate solution =

M1=Molarity of zinc sulfate solution=

 n_1 = number of moles of zinc sulfate = 1

EDTA

 V_2 =Volume of EDTA solution = ml

 M_2 =Molarity of EDTA solution = ?

 n_1 = number of moles of EDTA = 1

Molarity of EDTA
$$M_2 = \underline{M_1 V_1}$$

 V_2
 $=$
 $=$ M

Step –3: Determination of CaO content in Cement sample:

10 ml of cement solution is pipetted out into a 250 ml conical flask. 20 ml of distilled water is added followed by 10 ml of 4M NaOH solution to the flask (NaOH acts as a masks other metal ions.). The flask is then thoroughly shaken for 2 minutes. 5 ml of glycerol (to get sharp end – point), 5 ml of diethylamine (to maintain the pH of 12–13) and a pinch of Patton - Reeder's indicator is added. The contents are then titrated with EDTA from the burette until wine red colored solution changes to blue. The readings are then tabulated and then percentage of CaO is calculated. NaOH acts as a masking agent to mask other metal ions.

S. No	Volume of cement	Burette readings		Volume of EDTA
	sample solution in ml	Initial	Final	solution rundown in ml

Test Sample

 V_1 =Volume of test sample solution =

M1=Molarity of total CaO =

 n_1 = number of moles of CaO = 1



EDTA

V₂=Volume of EDTA solution = ml M₂=Molarity of EDTA solution = n_1 = number of moles of EDTA = 1

$$\frac{V_1 M_1}{n_1} X \frac{V_2 M_2}{n_2}$$
$$\therefore M_1 = \frac{V_2 M_2}{n_2} \times \frac{n_1}{V_1}$$

Concentration of the total CaO in test sample solution = M.

We know,

$$Molarity of Cao = \frac{weight of CaO}{Gram Mol.Weight of CaO} \times \frac{1000}{Volume in mL}$$

$$Weight of Ca0 = \frac{Molarity of Ca0 \times GMW of Ca0 \times Volume of Test sample in mL}{1000}$$

Percentage of Total Calcium Oxide:

% of Total Ca0 =
$$\frac{Weight of Ca0 obtained}{Weight of cement sample taken} \times 100$$

S. No	Given Amount	Reported Amount	% Error	Signature of the Faculty