

# **ENGINEERING CHEMISTRY LAB STUDENT LAB MANUAL**

For  
**(ECE and EEE)**

**Dr. Malla Balakrishna, M.Sc, Ph.D**  
Associate Professor in Chemistry

**Dr. Pondala Seetha Ram, M.Sc, Ph.D**  
Assistant Professor in Chemistry

**Mr. B.G.G. Krishnam Raju, M.Sc, (Ph.D)**  
Assistant Professor in Chemistry



**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 JNTUK, Kakinada)  
Jonnada (Village), Denkada (Mandal), Vizianagaram District,  
Andhrapradesh– 535 005

### **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).

3. Never taste any chemicals (you should never taste anything in the lab).
4. 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.

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 ECE DEPARTMENT**

### **Vision of the Department**

- Emerge as a Centre of Eminence in Electronics and Communication Engineering to impart quality education towards competent and skilled engineers.

### **Mission of the Department**

- Offering an inspiring and conducive learning environment to prepare skilled and competent engineers by having good infrastructure, laboratory facilities, and effective teaching-learning process.
- Fostering culture to face complex technological challenges through Internships, Projects and Industry-Institute Interactions in order to enhance employability skills.
- Creating an environment for higher studies and entrepreneurship by way of imparting quality education and promoting research activities.
- Imparting professional behavior and strong ethical values towards societal issues by encouraging socially relevant activities.



# **VISION & MISSION OF THE EEE DEPARTMENT**

**Vision of the Department**

- To be a hub for imparting knowledge, skills, and behaviour for exemplary contributions in the field of Electrical and Electronics Engineering.

**Mission of the Department**

- To impart Technical Education through the state-of-the-art infrastructure facilities, laboratories and instruction.
- To inculcate industry oriented learning through industrial visits, internships, projects at Industries, MOUs, to make students' technically skills oriented.
- Creating conducive environment for higher education, employment and entrepreneurship through quality education, professional skills and research.
- To promote societal commitment among students by inculcating moral and ethical values.

# **PROGRAM OUTCOMES (POs)**

## 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)**

## **ECE PSOs**

**PSO1:** Capable of design, develop, test, verify and implement analog and digital electronics and communication engineering systems and products.

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

## **EEE PSOs**

**PSO1:** Capable of design, develop, test, verify and implement electrical and electronics engineering systems and products.

**PSO2:** Succeed in national and international competitive examinations for successful higher studies and employment

# **ENGINEERING CHEMISTRY LAB SYLLABUS**



## **ENGINEERING CHEMISTRY LAB/ SEM-I (For ECE and EEE only)**

### **I YEAR –I SEMESTER SYLLABUS**

#### **COURSE OBJECTIVES**

- To familiarize the students with the basic concepts of Engineering Chemistry lab.
- To train the students on how to handle the instruments.
- To demonstrate the digital and instrumental methods of analysis.
- To expose the students in practical aspects of the theoretical concepts.

#### **COURSE OUTCOMESs**

- Explain the functioning of the instruments such as Conductivity and pH meters. (L-2)
- Interpret the graphical values to analyze the experimental results. (L-2)
- Determine the concentrations of Acid, Zinc and Copper. (L-5)
- Prepare polymers and nano materials. (L-4)
- Identify the safety precautions to carry out the experiments in the laboratory using chemicals. (L-3)

#### **LIST OF EXPERIMENTS**

1. Preparation of Phenol-Formaldehyde resin
2. Preparation of Urea-Formaldehyde resin
3. Determination of Zinc by EDTA method.
4. Determination of Copper in a copper ore
5. Determination of Corrosion of a metal in the presence/absence of inhibitors
6. Determination of Sulphuric acid in lead-acid storage cell
7. Determination of Hardness of a ground water sample.
8. Determination of strength of an acid by Conductometric method/pH metric method

#### **Virtual Labs**

9. Chemical Etching of Printed Circuit Boards(PCB)
10. Preparation of nano particles using sol-gel method.

#### **Text Books:**

1. Mendham J, Denney RC, Barnes JD, Thosmas M and Sivasankar B Vogel's Quantitative Chemical Analysis 6/e, Pearson publishers (2000).
2. N.K Bhasin and Sudha Rani Laboratory Manual on Engineering Chemistry 3/e, Dhanpat Rai Publishing Company (2007).

#### **INDEX**

Expt. No.	Experiment	CO's	Page No.
00	Introduction to chemistry lab		1-5
01	Preparation of Phenol-Formaldehyde resin		6-7
02	Preparation of Urea-Formaldehyde resin		8-9
03	Determination of Zinc by EDTA method.		10-12
04	Determination of Copper in a copper ore		13-15
	Determination of Sulphuric acid in lead-acid storage cell		16-17
06	Determination of Hardness of a ground water sample.		18-20
07	Determination of strength of an acid by Conductometric method/pH metric method		21-24
08	Determination of strength of an acid by pH metric method		25-28
09	Chemical Etching of Printed Circuit Boards(PCB)		29
10	Preparation of nano particles using sol-gel method.		30

## CO-PO'S MAPPING (ECE)

**COURSE OUTCOMES VS POs MAPPING (DETAILED; HIGH: 3; MEDIUM: 2; LOW: 1):**

PO CO	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12	PSO1	PSO2
CO1	2								2		1	1		
CO2	2	1							2			1		
CO3	2	1		1					2			1		
CO4	2			1					2		1	1		
CO5	2		1			1	1		2		1	1		
*BS1102	2	1	1	1		1	1		2		1	1		

*\* For Entire Course, PO & PSO Mapping*

## (EEE)

**COURSE OUTCOMES VS POs MAPPING (DETAILED; HIGH: 3; MEDIUM: 2; LOW: 1):**

PO CO	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12	PSO1	PSO2
CO1	2								2		1	1		
CO2	2	1							2			1		
CO3	2	1		1					2			1		
CO4	2			1					2		1	1		
CO5	2		1			1	1		2		1	1		
*BS1102	2	1	1	1		1	1		2		1	1		

*\* For Entire Course, PO & PSO Mapping*

## 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.

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**

\*\*\*\*\*

## 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.

1. **Qualitative Analysis:** It deals with identification and conformation of the nature of a substance present in a given sample.
2. **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:** 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 sulphate, sodium carbonate etc.

**Secondary Standards:** 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, KMnO<sub>4</sub>, HCl, H<sub>2</sub>SO<sub>4</sub> 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.

### Percent of Error:

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

$$\% \text{ of Error} = \frac{[\text{Actual value} - \text{Measured value}]}{\text{Actual value}} \times 100$$

### 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:

1. Molarity : It is the no of gram molecules of solute present in one liter of solution

$$\text{Molarity } M = \frac{\text{weight of the substance}}{\text{Gram molecular weight}} \times \frac{1000}{\text{volume in ml}}$$

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

$$\text{Normality } N = \frac{\text{weight of the substance} \times 1000}{\text{Gram equivalent weight} \times \text{Volume in ml}}$$

$$\text{Equivalent weight of an acid} = \frac{\text{molecular weight of an acid}}{\text{Number of replaceable } H^+ \text{ ions}}$$

$$\text{Equivalent weight of a base} = \frac{\text{molecular weight of a base}}{\text{Number of replaceable } OH^- \text{ ions}}$$

$$\text{Equivalent weight of an oxidizing or reducing agent} = \frac{\text{molecular weight of a substance}}{\text{No. of electrons gained or loosed}}$$

3. Molality: It is defined as the number of the moles of the solute present in 1 kg or 1000ml of the solvent,

$$\text{Molality } m = \frac{\text{no. of moles of solute}}{\text{Gram molecular weight}} \times \frac{1000}{\text{volume in ml}}$$

### Types of Volumetric Titrations:

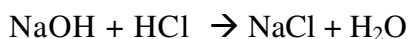
- 1) Acid-Base Titrations
- 2) Redox titrations
- 3) Complexometric Titrations

### 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.



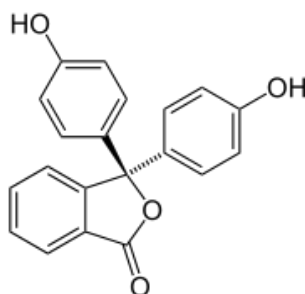
An indicator is (often) a weak acid or weak base that is placed into the unknown solution to determine the endpoint of the titration.

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 ( $\text{H}^+$ ) attached**

**pink in basic media ( $\text{H}^+$ ) ion is removed**



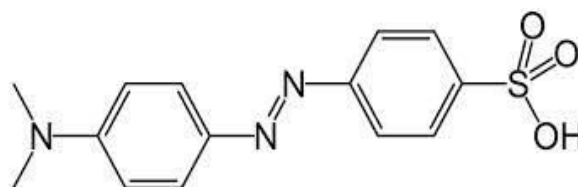
**Structure of phenolphthalein indicator**

**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 ( $\text{H}^+$ ) attach**

**yellow color in basic media ( $\text{H}^+$ ) removed**



**Structure of Methyl orange indicator**

## 2) 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

**1. Permanganometry:**  $\text{KMnO}_4$  is the oxidizing agent and titrated against a reducing agent like  $\text{Fe}^{2+}$  (ferrous ammonium sulphate) etc.

**2. Dichrometry:**  $\text{K}_2\text{Cr}_2\text{O}_7$  is the oxidizing agent and titrated against a reducing agent like  $\text{Fe}^{2+}$  (ferrous ammonium sulphate) etc.

**3. 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.

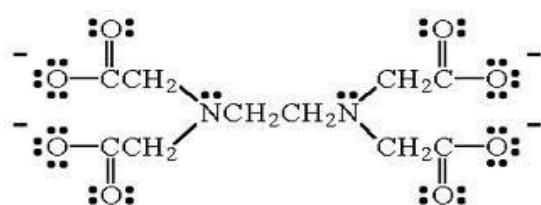
Eg: potassium permanganate. Such Indicators are called self indicators. Self indicators generally are strongly colored as a result of charge transfer transitions in them.

**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.

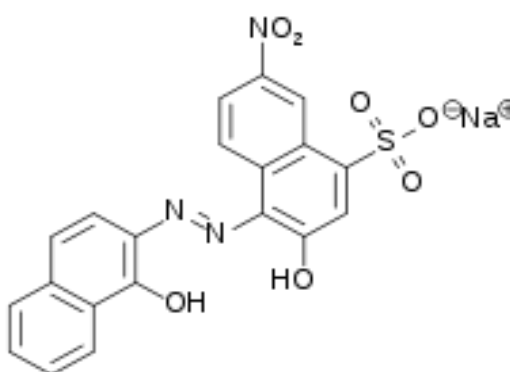
**External indicator :** In case a suitable redox indicator is not available for a given system, an indicator may be employed which will indicate the completion of reaction by physically or chemically reacting with the analyte (not through redox reaction). This reaction between indicator and the analyte 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.

**3) 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**



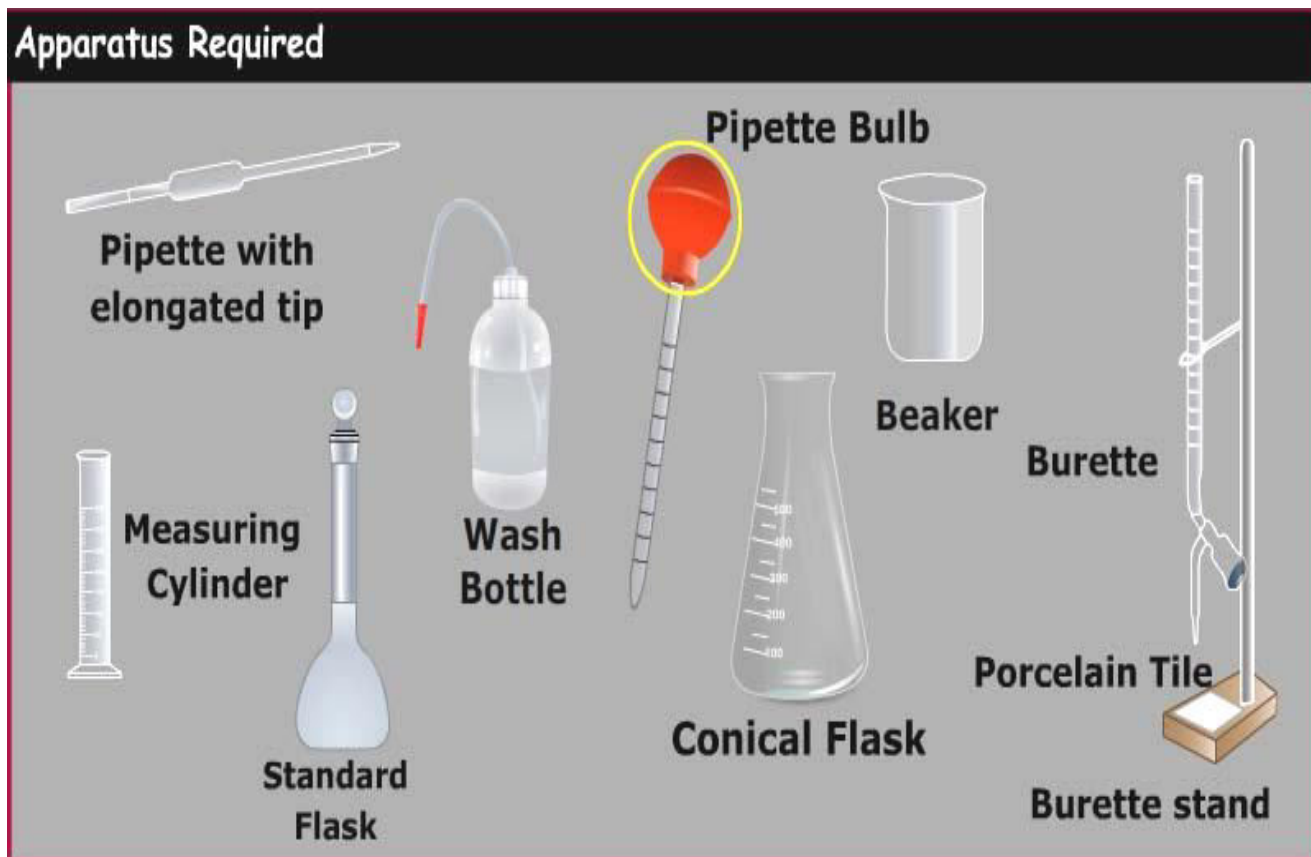
**Structure of EBT**

**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.



## EXPERIMENT-1

### PREPARATION OF PHENOL-FORMALDEHYDE RESIN (BAKELITE)

**Aim:** To prepare the phenol – formaldehyde resin

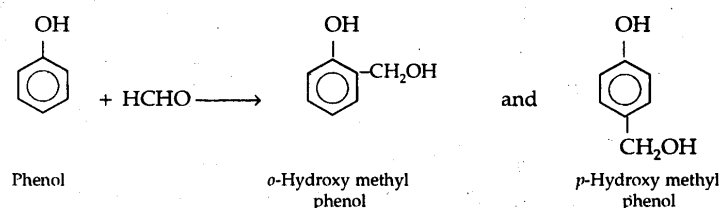
**Principle:** Phenol formaldehyde resins (PF) include synthetic thermosetting resins such as Bakelite obtained by the reaction of phenols with formaldehyde. Phenol-formaldehyde resins are formed by a step-growth polymerization reaction that can be either acid- or base-catalysed.

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 novolac 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 is less than one. Hexamethylene tetramine or "hexamine" is a hardener that is added to crosslink novolac. At  $\geq 180^\circ\text{C}$ , the hexamine forms crosslinks 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

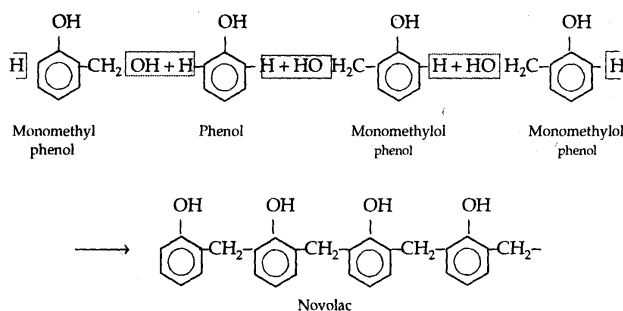
**Procedure:**

- 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 an oven
- Now the sample is weighed and its weight is reported

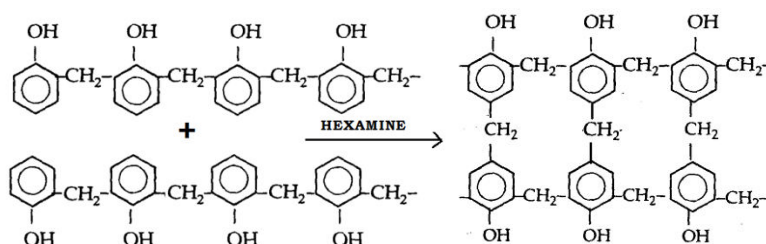
### Step-1: formation of o, p methyl phenol:



### Step-2: formation of novolac:



### 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

#### **Report:**

Amount of Resin formed	Marks awarded	Sign. of the faculty

**EXPERIMENT-2****PREPARATION OF UREA-FORMALDEHYDE RESIN**

**Aim:** - To prepare Urea-formaldehyde resin.

**Chemicals used:** - 40% formaldehyde, Urea, Conc. sulphuric acid, distilled water

**Apparatus required:** - Beakers, glass rod, funnel, measuring cylinder, filter paper.

**Principle:** - Urea formaldehyde resin is prepared by the condensation reaction between urea and formaldehyde in neutral or acidic conditions.

**Thermosets:** - The polymers which on heating change irreversibly into hard rigid and infusible materials are called thermosetting polymers. These polymers are usually prepared by heating relatively low molecular mass, semi fluid polymers, which becomes infusible and form an insoluble hard mass on heating. The hardening on heating is due to the formation of extensive cross-linking between different polymeric chains. This lead to the formation of a three dimensional network connecting the polymer chains. Since the 3D network structure is rigid and does not soften on heating, the thermosetting polymers cannot be reprocessed. Some important examples of thermosetting polymers are Phenol-Formaldehyde resin and Melamine Formaldehyde resins.

**Properties:-**

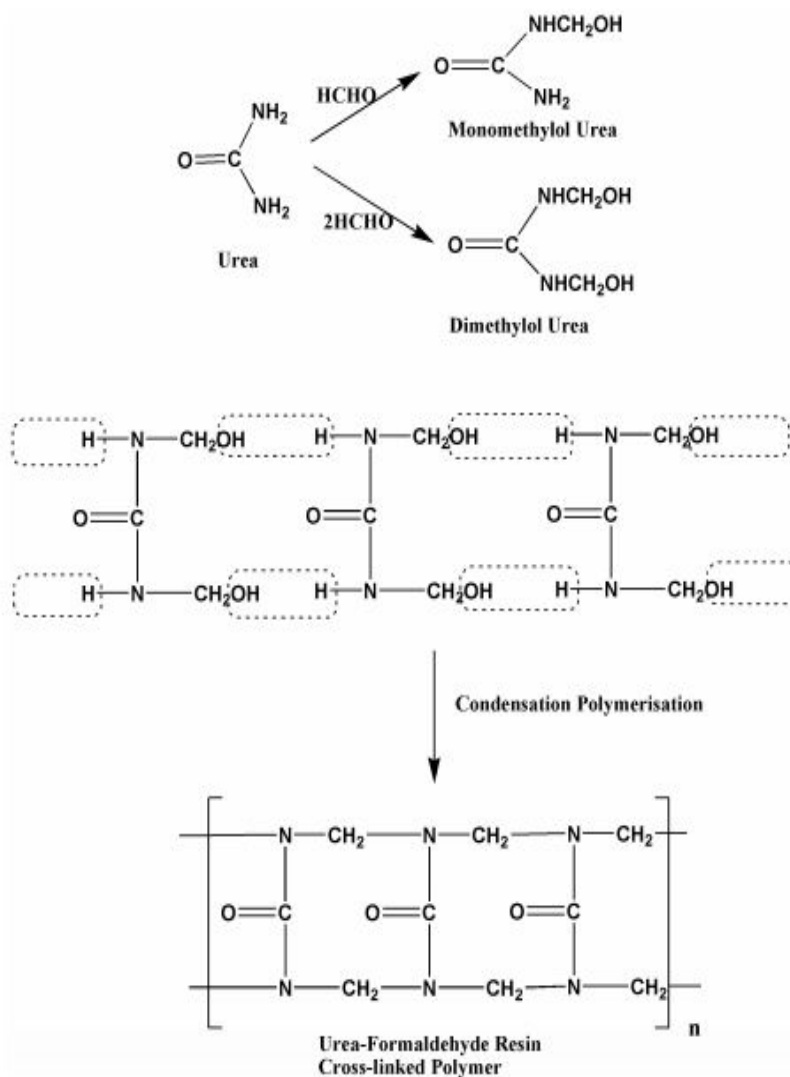
- They are clear and white.
- Better hardness and tensile strength than PF resins.
- Resistant to most of the solvents and grease.
- Excellent abrasion resistant and stable to light.
- Good adhesive characteristics.
- Good electrical insulators and possess chemical resistance.
- They can be synthesized in any desired color by adding proper pigment and filler during synthesis

**Uses:-**

- These resins are widely used in manufacture of buttons, bottle caps, house hold appliances, surgical items etc.
- They are used as adhesives in plywood industries.
- They are also used in the manufacturing of enamels and other surface coatings.
- Used for the finishing of cotton textiles (They impart stiffness, crease resistance, fire retardation, water repellency. They are also helpful for shrinkage control)

**Preparation:** - Urea and formaldehyde react with each other in neutral or acidic conditions to

give mono and dimethylol urea, which undergo further condensation reaction to give linear, partially cross linked or fully cross-linked polymer.



### Procedure:-

1. Place about 5ml of 40% formaldehyde solution in 100 ml beaker.
2. Add about 2.5 g of urea with constant stirring till saturated solution is obtained.
3. Add a few drops of conc. Sulphuric acid with constant stirring.
4. A voluminous white solid mass appears in the beaker.
5. Wash the white solid with water and dry it in the folds of filter paper.
6. Calculate the yield of the product

**Result: -** The yield of urea formaldehyde resin is \_\_\_\_ g.

**EXPERIMENT-3****DETERMINATION OF ZINC USING STANDARD EDTA SOLUTION**

**Aim:-** a) Standardization of the EDTA solution using standard Zinc sulphate solution.

a) Determination of zinc using standard EDTA.

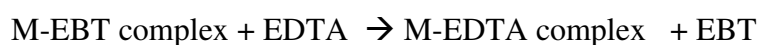
**Apparatus:-** Burette, pipette, burette stand, glazed tile, conical flask,

**Chemicals required:-** 0.1M Standard zinc sulphate solution, EDTA solution, Ammonia buffer, Erichrome Black T indicator.

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



Wine -red



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  $P^H=7$  to  $11$  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:-**

a) **Standardize the EDTA solution using standard zinc sulphate 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 sulphate 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 sulphate, 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.



S.No	Volume of Zinc Sulphate solution ( $V_1$ ) in ml	Burette readings		Volume of EDTA solution rundown ( $V_2$ ) in ml
		Initial	Final	

**Calculations:-**

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

**Zinc sulphate**

Volume of zinc sulphate solution  $V_1 = 20.0$  ml

Molarity of zinc sulphate solution  $M_1 = 0.1M$

**EDTA**

Volume of EDTA solution  $V_2 =$  ml

Molarity of EDTA solution  $M_2 = ?$

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

=

= M

**b) 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 sulphate 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 sulphate, 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.

S.No	Volume of Zinc Sulphate solution ( $V_3$ ) in ml	Burette readings		Volume of EDTA solution rundown ( $V_4$ ) in ml
		Initial	Final	

### Calculations:-

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

### Zinc sulphate

Volume of zinc sulphate solution  $V_3 = 20.0$  ml

Molarity of zinc sulphate solution  $M_3 = 0.1M$

### EDTA

Volume of EDTA solution  $V_4 =$

Molarity of EDTA solution  $M_4 =$

Molarity of EDTA  $M_3 = \frac{M_4 V_4}{V_3}$

=

Amount of zinc ion present in 100 ml of the given unknown solution =  $M_3 \times \frac{65.38}{1000} \times 100$  g

### Result:-

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

S.No	Given	Obtained	% of error = $\frac{\text{Given}-\text{Obtained}}{\text{Given}} \times 100$

**EXPERIMENT-4****DETERMINATION OF COPPER PRESENT IN A COPPER ORE**

**Aim:** a) Standardization of the EDTA solution using standard Zinc sulphate solution.

b) Determine the amount of copper present in a copper ore sample by using E.D.T.A

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

**Chemicals required:** - 0.1M zinc sulphate 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



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  $P^H=7$  to  $11$  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:-**

**a) Standardize the EDTA solution using standard zinc sulphate 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 sulphate 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 sulphate, 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.

S.No	Volume of Zinc Sulphate solution ( $V_1$ ) in ml	Burette readings		Volume of EDTA solution rundown ( $V_2$ ) in ml
		Initial	Final	

**Calculations:-**

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

**Zinc sulphate**

Volume of zinc sulphate solution  $V_1 = 20.0$  ml

Molarity of zinc sulphate solution  $M_1 =$

**EDTA**

Volume of EDTA solution  $V_2 =$

Molarity of EDTA solution  $M_2 =$

$$\text{Molarity of EDTA } M_2 = \frac{M_1V_1}{V_2}$$

**b) Determination of the amount of copper present in a given copper ore 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:** - Conical 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.

S.No	Volume of Copper Sulphate solution ( $V_3$ ) in ml	Burette Readings		Volume of EDTA solution rundown ( $V_2$ ) in ml
		Initial	Final	

**Calculations:-**

The copper ion can be determined by using the following formula:  $M_2V_2 = M_3V_3$

**Copper sulphate**

Volume of copper sulphate solution  $V_3 = 20$  ml

Molarity of copper sulphate solution  $M_3 = ?$

**EDTA**

Volume of EDTA solution  $V_2 =$  ml

Molarity of EDTA solution  $M_2 =$  M

The Molarity of copper sulphate solution,  $M_3 = \frac{M_2V_2}{V_3}$

$$= M$$

The amount of copper ion present in a given unknown solution

$$= \frac{M_3 \times \text{gram atomic weight of Cu}^{+2} (63.54) \times 100}{1000}$$

1000

$$= \text{gm}$$

**Result:** - The amount of copper ion present in given 100ml solution = gm.

**Report:**

S.No	Given	Obtained	% of error: $\frac{\text{Given}-\text{Obtained}}{\text{Given}} \times 100$

**Precautions:**

1. Pippetting as to be accurate in order to avoid excess addition of the titrating agent.
2. Reading should be taken to avoid the parallax error.
3. The titration should be placed on white paper to identify properly the color change at the end point.
4. All the glass apparatus should be washed thoroughly with distilled water before use.
5. They should not be any leakage in the burette.

### EXPERIMENT-5

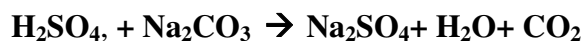
#### DETERMINATION OF SULPHURIC ACID IN LEAD ACID STORAGE CELL

**Aim:** - Determine the amount of  $\text{H}_2\text{SO}_4$  present in lead-acid storage cell using 0.1N  $\text{Na}_2\text{CO}_3$

**Apparatus:** - Burette, pipette, burette stand, conical flask, wash bottle, glazed tile

**Chemicals required:** -  $\text{H}_2\text{SO}_4$ , 0.1N  $\text{Na}_2\text{CO}_3$ , distilled water, Methyl orange.

**Principle:** -  $\text{H}_2\text{SO}_4$ , reacts with  $\text{Na}_2\text{CO}_3$  the following manner



$\text{Na}_2\text{CO}_3$  is a weak base; it can be estimated by titrating with a strong acid like  $\text{H}_2\text{SO}_4$  using methyl Orange as indicator

**Procedure:-**

b) **Determination of  $\text{H}_2\text{SO}_4$  using standard solution of 0.1N  $\text{Na}_2\text{CO}_3$**

**Burette:** - The burette is cleaned first with distilled water and filled with  $\text{H}_2\text{SO}_4$ , without any air bubbles.

**Conical Flask:** - Conical flask is washed with distilled water and then pipette out 20ml of  $\text{Na}_2\text{CO}_3$  solution into the conical flask and add 2 or 3 drops of Methyl Orange indicator.

**Indicator:** - Methyl Orange

**End Point:** - yellow to red

The conical flask is placed under the burette on a glazed tile, then the  $\text{H}_2\text{SO}_4$ , present in the burette is slowly rundown by shaking the conical flask in clockwise direction continuously, the titration is continued until a color change yellow to red which is the endpoint. The experiment is repeated till the concurrent readings are obtained.

S.No	Volume of $\text{Na}_2\text{CO}_3$ Solution in ml	Burette Readings		Volume of $\text{H}_2\text{SO}_4$ rundown in ml (b-a)
		Initial (a)	Final (b)	

**Calculations:-**

Normality of  $\text{H}_2\text{SO}_4$ , is calculated using the equation

$$N_1 V_1 = N_2 V_2$$

$\text{Na}_2\text{CO}_3$

Normality of  $\text{Na}_2\text{CO}_3$   $N_1 = 0.1\text{N}$

Volume of  $\text{Na}_2\text{CO}_3$  solution  $V_1 = 20\text{ml}$



Normality of  $\text{H}_2\text{SO}_4$   $N_2 =$

Volume of  $\text{H}_2\text{SO}_4$   $V_2 =$  ml (burette readings)

Normality of  $\text{H}_2\text{SO}_4$  ( $N_2$ ) =  $N_1 V_1 / V_2$

=

= N

**Report:-**

The amount of  $\text{H}_2\text{SO}_4$  present in lead-acid storage cell = gm

**Precautions:**

1. Pipetting as to be accurate in order to avoid excess addition of the titrating agent.
2. Excess of indicator should not be used.
3. Reading should be taken to avoid the parallax error.
4. The titration should be placed on white paper to identify properly the color change at the end point.
5. All the glass apparatus should be washed thoroughly with distilled water before use.
6. They should not be any leakage in the burette.

**EXPERIMENT-6****DETERMINATION OF HARDNESS OF A GROUND WATER SAMPLE****Aim:-**

- a) Standardization of the EDTA solution using standard Zinc sulphate solution.
- b) Determination of the temporary and permanent hardness of given water sample.

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

**Chemicals required:** - 0.1M Standard zinc sulphate solution, EDTA solution, Ammonia buffer, Erichrome Black T indicator.

**Principle:-**

Metal ions form a complex with EDTA according to the equation



Wine -red



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  $P^H=7$  to  $11$  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:-**

- b) **Standardize the EDTA solution using standard zinc sulphate 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 sulphate 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 sulphate, 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 Sulphate solution ( $V_1$ ) in ml	Burette readings		Volume of EDTA solution rundown ( $V_2$ ) in ml
		Initial	Final	
1				
2				
3				

### Calculations:-

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

### Zinc sulphate

Volume of zinc sulphate solution  $V_1 = 20.0$  ml

Molarity of zinc sulphate solution  $M_1 = 0.1N$

### EDTA

Volume of EDTA solution  $V_2 =$  ml

Molarity of EDTA solution  $M_2 =$  M

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

$$= \text{M}$$

### b) 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 sulphate 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.

S.No	Volume of Water Sample ( $V_1$ ml)	Burette readings		Volume of EDTA solution rundown ( $V_2$ ) in ml
		Initial	Final	

**Calculation:**

The concentration of water solution can be calculated from the equation,  $V_1 M_1 = V_2 M_2$

Water sample

$V_3$  = Volume of Water sample (20.0ml)

$M_3$  = Molarity of water sample (?)

EDTA

$V_2$  = Volume of EDTA Solution

$M_2$  = Molarity of EDTA Solution

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

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

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

$$= M_3 \times 100 \times 1000 \text{ mg/lit or ppm}$$

$$= \quad \text{ppm}$$

**Result:**

Total hardness present in the given water sample =  $\quad$  ppm

**Precautions:**

1. Pipetting as to be accurate in order to avoid excess addition of the titrating agent.
2. Reading should be taken to avoid the parallax error.
3. The titration should be placed on white paper to identify properly the color change at the end point.
4. All the glass apparatus should be washed thoroughly with distilled water before use.
5. They should not be any leakage in the burette.

## EXPERIMENT-7

### DETERMINATION OF STRENGTH OF AN ACID BY CONDUCTOMETRIC METHOD

**Aim:** To determine the amount of unknown acid solution with standard base solution by conductometric method.

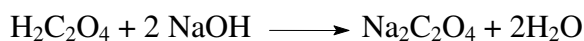
**Apparatus:** Conductivity meter (with cell), burette (10ml), volumetric flask (100 ml), beakers (100 ml), stirrer / glass rod.

**Chemicals:** Stock acid solution, 0.05 M oxalic acid in 100ml volumetric flask and Stock base solution.

**Principle:** Conductometric titrations work on the principle of Ohm's law. As current is inversely proportional to Resistance (R) and the reciprocal of resistance is termed as Conductance, and its unit is Siemen (mho)  $\text{cm}^{-1}$ . The electrical conductivity of a solution depends on the number of ions and their mobility. In Conductometric titrations, the titrant is added from the burette, and the conductivity readings are plotted against the volume of the titrant. Upon adding a strong base to the strong acids, the conductance falls until the strong acid is neutralized then raised. Such a titration curve consists of 2 lines which intersect at a particular point, known as the **End point or Equivalence point**. The method can be used for titrating coloured solutions or homogeneous, which cannot be used with normal indicators.

#### Strong Acid with a Strong Base:

For example, in the titration of HCl versus NaOH, the addition of a strong base (NaOH) to a strong acid (HCl). Before NaOH is added, the conductance is high due to the presence of highly mobile hydrogen ions. When the base is added, the conductance falls due to the replacement of hydrogen ions by the added cation as  $\text{H}^+$  ions react with  $\text{OH}^-$  ions to form undissociated water. This decrease in the conductance continues till the equivalence point. At the equivalence point, the solution contains only NaCl. After the equivalence point, the conductance increases due to the large conductivity of  $\text{OH}^-$  ions.



Formula:

$$\frac{M_1 V_1}{n_1} = \frac{M_2 V_2}{n_2} \quad \frac{M_2 V_2}{n_2} = \frac{M_3 V_3}{n_3}$$

#### Procedure:

##### Step 1: Standardization of sodium hydroxide by using oxalic acid

1. Rinse and fill the burette with the given NaOH solution
2. Pipette out 20 ml of 0.05 M oxalic acid solution into a clean conical flask
3. Add 1 or 2 drops of phenolphthalein indicator to oxalic acid solution.
4. Titrate the solution against sodium hydroxide solution drop wise with shaking till the solution changes to pale pink
5. Note the volume of NaOH used. It is the end point.
6. Repeat the titration until the concordant readings are obtained
7. Calculate the molarity of NaOH by using the formula mentioned above

### Step-1: Observations & Calculations:

Burette : NaOH Solution  
 Conical Flask : 20 ml Oxalic Acid  
 Indicator : Phenolphthalein  
 End Point : Colour less to Pale pink.

S.No.	Volume of Oxalic acid ( $v_1$ ml)	Burette readings (ml)		Volume of NaOH Rundown ( $v_2$ ml)
		Initial	Final	
1				
2				
3				
4				

### Calculations:

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

### Oxalic Acid:

$M_1$  = Molarity of Oxalic Acid = 0.05M

$V_1$  = Volume of Oxalic Acid = 20 ml

$n_1$  = Moles of Oxalic Acid = 1 mole

### Sodium Hydroxide:

$M_2$  = Molarity of NaOH = ?

$V_2$  = Volume of NaOH =

$n_2$  = Moles of NaOH = 2 mole

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

Molarity of NaOH = ----- M

**Step 2: Determination of molarity of unknown HCl by using standard NaOH through conductometric titration**

1. In 100 ml beaker take 25ml of given unknown HCl solution and add 25ml of distilled water.  
The contents are shaken thoroughly.
2. Now, the conductivity cell is immersed in the beaker and the initial conductance of the solution is taken by stirring the solution and keeping it constant.
3. Then, 0.5 ml portions of base is added from the burette and stirred well. The conductance of the solution for each addition is to be noted.
4. The conductivity is corrected by multiplying with the factor  $[(v+V)/V]$ , where 'v' is the volume of base added and 'V' is the volume of solution initially taken in the beaker
5. Plot the graph with respect to the volume of base consumed versus corrected conductance.  
From the intersection point on the graph which gives value represents the equivalence points of acid and base.

**Observations and Calculations:** Conductometric titration in between HCl and NaOH

Volume of base added	Conductance	Corrected conductance $C^1 = C[(v+V)/V]$

**Calculation of Unknown molarity of HCl solution:**

$$\frac{M_2 V_2}{n_2} = \frac{M_3 V_3}{n_3}$$

**Sodium Hydroxide:**

$M_2$  = Molarity of NaOH =

$V_2$  = Volume of NaOH =

$n_2$  = Moles of NaOH = 1 mole

**HCl:**

$M_3$  = Molarity of HCl =?

$V_3$  = Volume of HCl = 25 ml

$n_3$  = Moles of HCl = 1 mole

$$M_3 = \frac{M_2 V_2 n_3}{n_2 V_3}$$

**Molarity of HCl = ----- M**

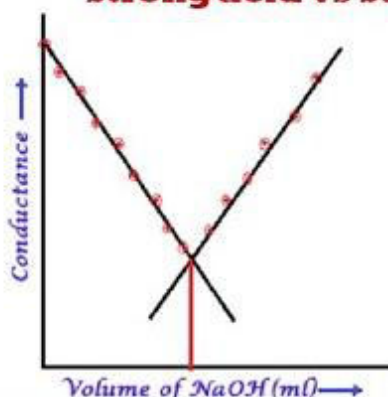
**Amount of HCl = ----- grs/l**

**Report:**

S.No	Given Amount of unknown Acid	Reported Amount of unknown Acid	% Error	Marks	Signature of the Faculty
1					

**Model Graph:**

**Conductometric titration:  
Strong acid vs Strong base**



❖ equivalence point/neutralisation point is determined graphically

**EXPERIMENT-8****DETERMINATION OF STRENGTH OF AN ACID BY pH METRIC METHOD**

**Aim:** To determine the Amount of unknown acid solution with standard base solution by pH metric method.

**Apparatus:** pH Meter, Glass membrane electrode, 100ml Beaker, Burette, Volumetric Flask, Glass Rod.

**Chemicals:** Stock acid solution, 0.2 M oxalic acid and Stock base solution.

**Principle:** When a glass surface is in contact with a solution it acquires a potential which depends on  $H^+$  ion concentration of solution. This observation which has been made by Haber is now used as basis of method of determining the pH of a solution where other electrode cannot be used. It has been observed that potential difference electrode has attained much attention in recent years because it can be used almost in all solutions except exists at the interface between glass and solution containing  $H^+$  ions. The magnitude of the difference of potential for a given variety of glass varies with its ions concentration at  $25^{\circ}C$  given by:

$E = E^0 + 0.0591 \log [H^+]$ ;  $E^0 = A$  constant for the given glass electrode.



Formula:

$$\frac{M_1 V_1}{n_1} = \frac{M_2 V_2}{n_2} \quad \frac{M_2 V_2}{n_2} = \frac{M_3 V_3}{n_3}$$

**Procedure:****Step 1: Standardization of sodium hydroxide by using oxalic acid**

1. Rinse and fill the burette with the given NaOH solution
2. Pipette out 20 ml of 0.2 M oxalic acid solution into a clean conical flask
3. Add 1 or 2 drops of phenolphthalein indicator to oxalic acid solution.
4. Titrate the solution against sodium hydroxide solution drop wise with shaking till the solution changes to pale pink
5. Note the volume of NaOH used. It is the end point
6. Repeat the titration until the concordant readings are obtained
7. Calculate the molarity of NaOH by using the formula mentioned above

**Step-1: Observations & Calculations:**

Burette : NaOH Solution  
 Conical Flask : 20 ml Oxalic Acid  
 Indicator : Phenolphthalein  
 End Point : Colour less to Pale pink.

S.No.	Volume of Oxalic acid (v <sub>1</sub> ml)	Burette readings (ml)		Volume of NaOH Rundown (v <sub>2</sub> ml)
		Initial	Final	
1				
2				
3				
4				

**Calculations:**

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

**Oxalic Acid:**

M<sub>1</sub> = Molarity of Oxalic Acid = 0.2M

V<sub>1</sub> = Volume of Oxalic Acid = 20 ml

n<sub>1</sub> = Moles of Oxalic Acid = 1

**Sodium Hydroxide:**

M<sub>2</sub> = Molarity of NaOH = ?

n<sub>2</sub> = Moles of NaOH = 2

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

Molarity of NaOH = M

**Step 2: Determination of molarity of unknown HCl by using standard NaOH through pH metric titration**

1. Rinse and fill the burette with standard NaOH solution
2. Take 5ml of given unknown HCl solution into 100ml beaker and add 45ml of distilled water.  
The contents are shaken thoroughly.
3. The glass membrane electrode is dipped into the beaker containing the solution.



- Initially at “0” Burette reading of NaOH solution, pH of the unknown HCl solution can be measured.
- Then 0.5 ml of base is added from the burette to the acid solution and on stirring thoroughly the pH of the resultant solution can be noted.
- The pH is noted every time by the addition of 0.5 ml base and finally you observe the pH jump is between  $V_1$  and  $V_2$  ml. After pH jump you need to note about 10 readings.
- Plot the graph with the volume of base on X - axis versus pH on Y-axis. Identify the suitable jump which changes the medium from acidic pH to Basic pH.
- Take the average in-between the jump values and draw a line which intercepts X axis. The intersection point gives value of the equivalence point (End point) of acid and base.

**Step-2: Observations and Calculations:** pH Metric titration in between **HCl** and **NaOH**

VOLUME OF NaOH ADDED	pH

Calculation of unknown molarity of HCl solution:

$$\frac{M_2 V_2}{n_2} = \frac{M_3 V_3}{n_3}$$

**Sodium Hydroxide:** $M_2$  = Molarity of NaOH = $V_2$  = Volume of NaOH = $n_2$  = Moles of NaOH = 1**HCl:** $M_3$  = Molarity of HCl = ? $V_3$  = Volume of HCl = 5ml $n_3$  = Moles of HCl = 1

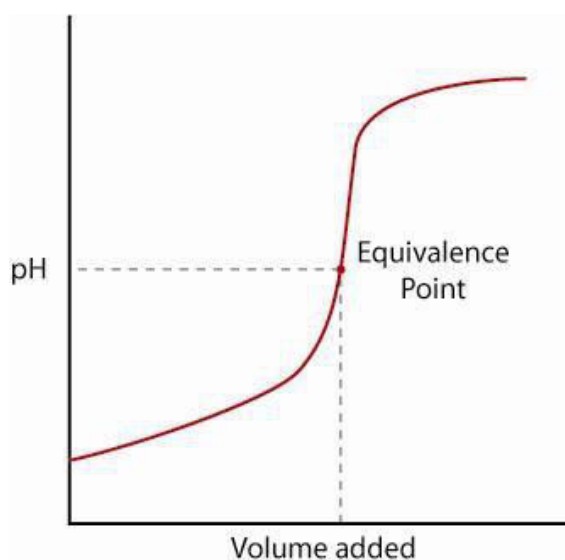
$$M_3 = \frac{M_2 V_2 n_3}{n_2 V_3}$$

Molarity of HCl = M

Amount of HCl = grs/l

**Report:**

S.No	Given Amount of unknown Acid	Reported Amount of unknown Acid	% Error	Marks	Signature of the Faculty
1					

**Model Graph:**

**EXPERIMENT-9****CHEMICAL ETCHING OF PRINTED CIRCUIT BOARD (PCB)**

**Introduction:** In this process, we hang the Copper clad sheet having the design of PCB printed on it with a Blue ink with the help of holder. So now after dipping the sheet in Ferric Chloride solution, the part of PCB which is covered with the Blue ink remains as it is but the part which has no ink on it, dissolved in the solution of Ferric Chloride after undergoing chemical reaction. Hence we are now left with the Printed Circuit Board as required; with the design printed on it in the form of Copper Tracks.

**Chemicals Required:** Ferric Chloride

**Procedure:**

- Start the etching machine heater, 15 minutes before using it so that the Ferric Chloride solution reaches and maintains itself at the operational temperature during etching and also the process takes less time.
- Put the clad in the etching machine and turn the timer on. This process should be made to happen for about 6-9 minutes or the user can check whether the etching has been done completely or not by removing the flap cover of the  $\text{FeCl}_3$  container.
- When the etching is done wash it with water, till the dye color (i.e. Blue Color) is removed. Hence now we have our Printed Circuit Board ready to be used.

**Report:**

**EXPERIMENT-10****PREPARATION OF NANOMATERIALS USING SOL-GEL/ PRECIPITATION  
METHOD.**

**Aim:** To prepare ZnO nano particles by precipitation method.

**Chemical required:** Zinc Nitrate, KOH and alcohol.

**Apparatus Required:** Centrifuge, large beaker and magnetic stirrer.

**Preparation:** Zinc nitrate as the precursor, KOH as a precipitating agent to synthesize ZnO nanoparticles were purchased from Sigma-Aldrich. Preparation ZnO nanoparticles were synthesized by direct precipitation method using zinc nitrate and KOH as precursors. In this work, the aqueous solution (0.2 M) of zinc nitrate ( $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) and the solution (0.4 M) of KOH were prepared with deionized water, respectively. The KOH solution was 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 min and washed three times with distilled water, and washed with absolute alcohol at last. The obtained product was calcined at 500 °C in air atmosphere for 3 hr.

**Report:**

Weight of the ZnO is formed \_\_\_\_\_ g