

# ENGINEERING CHEMISTRY

# (Laboratory Manual) 2022-2023

By

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> Common To All Branches

- The student is expected to learn from this laboratory course the concept of error and its analysis.
- It also allows the student to develop experimental skills to design new experiments in Engineering.
- With the exposure to these experiments the student can compare the theory and correlate with experiment.

# ENGINEERING CHEMISTRY LAB PROGAMME OUT COME:

- To apply the knowledge on mathematics, science and engineering concepts in complex engineering problems
- To perform investigations, design as well as conduct experiments, analyze and interpret the results to provide valid conclusions
- Perform effectively as a member/leader in multidisciplinary teams
- Identify, formulate and solve complex problems to achieve demonstrated conclusions
- Acquire Knowledge of contemporary issues
- Ability to choose & apply the techniques, skills, and modern engineering tools necessary for engineering practice.
- An ability to design, analyze, construct and synthesize circuits related electronics and communication engineering

# **INSTITUTION VISION AND MISSION**

# **VISION**

To establish as an ideal academic institutions in the service of the nation, the world and the humanity by graduating talented engineers to be ethically strong, globally competent by conducting high quality research, developing breakthrough technologies, and disseminating and preserving technical knowledge.

# **OUR MISSION**

To fulfill the promised vision through the following strategic characteristics and aspirations:

• Contemporary and rigorous educational experiences that develop the engineers and managers.

**O** An atmosphere that facilitates personal commitment to the educational success of students in an environment that values diversity and community.

**O** Undergraduate programs that integrate global awareness, communication skills and team building.

**O** education and training that prepares students for interdisciplinary engineering research and advanced problem solving

#### **COURSE OBJECTIVES**

The course consists of experiments related to the principles of chemistry required for engineering student. The student will learn:

- Estimation of hardness of water to check its suitability for drinking purpose.
- Students are able to perform estimations of acids and bases using conductometry, potentiometry methods.
- Students will learn to prepare polymers such as Bakelite and Thiokol rubber in the laboratory.
- Students will learn skills related to the lubricant properties such as saponification value, surface tension and viscosity of oils.

# **Course Outcomes**

The experiments will make the student gain skills on:

- Determination of parameters like hardness of water
- Able to perform methods such as conductometry, potentiometry and in order to find out the concentrations or equivalence points of acids and bases.
- Students are able to prepare polymers like bakelite and Thiokol rubber.
- Estimations saponification value, surface tension and viscosity of lubricant oils.





Head of the Department

Lecture In-Charge

**External Examiner** 

**Internal Examiner** 

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# PREFACE

This book entitled "Engineering chemistry Lab Manual" is intended for the use of First year B.Tech students of Marri Laxman Reddy Institute of Technology and Management, Dundigal, Hyderabad. The main objective of the Engineering chemistry Lab Manual is to furnish the conceptual understanding of the basic principles. The experiments are selected from various areas of chemistry like water treatment, viscosity, preparation of rubbers like bakelite and Thiokol rubber, surface tension, e.t.c. The book was written as per the new syllabus prescribed by the JNTUH University in a simple language. Viva voice questions also included in the book. These experiments will help the students to expertise in the analysis of various concepts . Hence we hope this book serve for better understanding by the student community with all details of experiments.

By, Z.T.Anitha Krupanidhi Asst.Professor, Department of Freshman engineering

#### ACKNOWLEDGEMENT

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By, Z.T.Anitha Krupanidhi Asst.Professor, Department of Freshman engineering

# Engineering Chemistry Laboratory

#### Chemistry Lab Do's & Don'ts

The chemistry laboratory must be a safe place in which to work and learn about chemistry. Most of these involve just using common sense.

- 1. Wear a chemical-resistant apron.
- 2. Be familiar with your lab assignment **before** you come to lab. Follow all written and verbal instructions carefully. Observe the safety alerts in the laboratory directions. If you do not understand a direction or part of a procedure, ask the teacher before proceeding.
- 3. When entering the lab/classroom, do not touch any equipment, chemicals, or other materials without being instructed to do so. Perform only those experiments authorized by the instructor.
- 4. No student may work in the laboratory without an instructor present. Work only with your lab partner(s). Do not venture to other lab stations for any reason.
- 5. Do not wear bulky or dangling clothing.
- 6. Never eat or drink in the laboratory. Don't chew on the end of a pen which was lying on the lab bench.
- 7. Wash acid, base, or any chemical spill off of yourself immediately with large amounts of water. Notify your teacher of the spill.
- 8. Clean up spills immediately. If you spill a very reactive substance such as an acid or base, notify the people in the area and then obtain assistance from your teacher. Acid spills should be neutralized with baking soda, base spills with vinegar before cleaning them up.
- 9. If chemical substances get in your eye, wash the eye out for 15 minutes. Hold your eye open with your fingers while washing it out.
- 10. When weighing never place chemicals directly on the balance pan. Never weigh a hot object.
- 11. Never smell anything in the laboratory unless your teacher tells you it is safe. Do not smell a substance by putting your nose directly over the container and inhaling. Instead, waft the vapors toward your nose by gently fanning the vapors toward yourself.
- 12. Do not directly touch any chemical with your hands. Never taste materials in the laboratory.
- 13. If you burn yourself on a hot object, immediately hold the burned area under cold water for 15 minutes. Inform your teacher.
- 14. Observe good housekeeping practices. Work areas should be kept clean and tidy at all times. Only lab notebooks or lab handouts should be out on the table while performing an Experiment. Books and book bags should not be on the lab table. Passageways need to be clear at all times.
- 15. Always replace lids or caps on bottles and jars.

#### 16. If your Bunsen burner goes out, turn the gas off immediately.

#### **1.ESTIMATION OF HARDNESS OF WATER BY EDTA METHOD**

#### **INTRODUCTION:**

Volumetric analysis is one of the quantitative methods of analysis which basically involves the determination of the quantity of a substance present in a given solution by reacting a known volume of it with a solution of another substance of known concentration.

The process by which this analysis is carried out is called 'titration'.

Hardness of water is determined by titrating with a standard solution of ethylene diamine tetra acetic acid (EDTA) which is a complexing agent. Since EDTA is insoluble in water, the disodium salt of EDTA is taken for this experiment. EDTA can form four or six coordination bonds with a metal ion

#### AIM:

To estimate the total hardness, permanent hardness and temporary hardness of water by using standard solution of EDTA

#### **APPARATUS:**

Burette, pipette, Conical flask, Beakers, Standard flask, Burette stand and funnel etc..

#### **CHEMICALS REQUIRED:**

Buffer sol<sup>n</sup>, EDTA sol<sup>n</sup>, Eriochrome black-T, Magnesium sulphate etc.

#### **PRINCIPLE:**

Hard water which contains  $Ca^{2+}$  and  $Mg^{2+}$  ions which forms wine red color complex with the indicator

 $Ca^{2+}$  (or)  $Mg^{2+} + EBT \longrightarrow Ca-EBT$  (or) Mg-EBT(Wine red colour complex) EDTA forms a colour less complex with the metal ions ( $Ca^{2+}$  and  $Mg^{2+}$ )

Ca-EBT (or) Mg-EBT + EDTA———> Ca-EDTA (or) Mg-EDTA + EBT (Wine red colour complex) (Colorless stable complex) (Blue) When free ions are not available, EDTA extracts the metal from (ion) metal ion indicator complex, there by releasing the free indicator.

#### **PROCEDURE:**

#### STEP-I

#### PREPARATION OF STANDARD SOLUTION OF MgSO4:

Weigh the approx 0.25gm of  $MgSO_4$  and transfer into 100ml standard flask through the funnel and dissolve in minimum quantity of distilled water. Make up the solution up to the mark with distilled water and shake the flask well for uniform concentration then calculate the Molarity of  $MgSO_4$ 

M. Wt. of  $MgSO_{4} = 246.48 gm$ 

Molarity of MgSO<sub>4</sub> (M1) =  $\begin{array}{c} 0.25 \\ - 246.48 \end{array} \begin{array}{c} 1000 \\ 100 \end{array} = 0.01 \end{array}$  =0.01 M1 = 0.01

#### **STEP-II**

#### STANDARDISATION OF EDTA SOLUTION:

Pipette out 20ml of MgSO<sub>4</sub> sol. into a clean conical flask. Add 2ml of buffer sol. and add 2 to 3 drops of EBT indicator and it gets wine red color sol. Take EDTA solution in a burette after titrate with EDTA solution till wine red color changes to blue color. Note the burette reading and repeat the titration to get concurrent values.

S No	Volume of MgSO <sub>4</sub> in	Burette	Reading	Volume of EDTA
5.110	ml (V1)	Initial	Final	consumed (V2)
1	20			
2	20			
3	20			

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

$$M2 = \frac{M1V1}{V2}$$

#### **STEP-III**

#### STANDARDISATION OF HARD WATER:

Pipette out 20ml of tap water into a 250ml conical flask add 2 ml of buffer sol. and add 2-3drops of EBT indicator. Titrate the wine red color sol. with EDTA taken in burette, till a blue color end point is obtained. Repeat the titration to get concurrent values.

S. No.	Volume of Hard	Burette Reading		Volume of EDTA
5. NU	water in(V3)	V3) Initial	Final	consumed (V2)
1	20			
2	20			
3	20			

 $M_3$  = molarity of hard water  $M_2$  = EDTA molarity  $V_3$  = volume of Hard water  $V_2$  = volume of EDTA consumed

$$M_3V_3 = M_2V_2$$

M2 V2  $M3 = \_____V3$ 

Total hardness=  $M_3X100X1000$  = -----PPM

#### **STEP-IV**

#### **STANDARDISATION OF PERMANENT HARDNESS OF WATER:**

Pipette out 100ml of hard water sample into a beaker containing 250ml and boil the water till volume reduces to 50ml (all the bicarbonates of  $Ca^{2+}$ ,  $Mg^{2+}$  decomposes to  $CaCO_3$  and  $Mg(OH)_2$  respectively). Cool the solution and filter the water into beaker then pipette out 20ml of this cool water sample in to 250ml conical flask add 2ml of buffer sol. and 2-3 drops of EBT indicator. Titrate the wine red color solution with EDTA taken in the burette, till a blue colored solution end point is obtained. Repeat the titration to get concurrent values.

S. No.	Volume of Hard	<b>Burette Reading</b>		Volume of EDTA
5. NO	water in (V4)	Initial	Final	consumed (V2)
1	20			
2	20			
3	20			

 $\begin{array}{ll} M_4 = molarity \mbox{ of hard water } & M_2 = EDTA \mbox{ molarity } \\ V_4 = volume \mbox{ of hard water } & V_2 = volume \mbox{ of EDTA consumed } \end{array}$ 

$$\begin{split} M_4 V_4 &= M_2 V_2 \\ M2 V2 \\ M4 &= ----- \\ V4 \end{split}$$

 $Permenent \ hardness = \ M_4 X100 X1000 \ = -----PPM$ 

# **RESULT:**

- 1) Total Hardness in given sample is \_\_\_\_\_ PPM
- 2) Permanent Hardness in given sample is \_\_\_\_\_ PPM
- 3) Temporary hardness in given sample is \_\_\_\_\_ PPM

#### **VIVA QUESTIONS**

- 1. What are complexometric titrations?
- 2. Name the most important complexing agent employed in complexometry ?
- 3. What are the chemicals used in edta experiment ?
- 4. What form of EDTA is used in titrametric analysis ?
- 5. Expand EDTA?
- 6. Give some examples of titrations involving EDTA as a complexing agents ?
- 7. Which type of ligand is EDTA?
- 8. What is the principle involved in this experiment ?
- 9. Name the most widely used indicator in EDTA titrations. How does it act ?
- 10. At what pH the hardness of water is estimated by EDTA method ?
- 11. How this pH is maintained ?
- 12. What is a buffer solution ?
- 13. What is the composition of buffer solution ?
- 14. Write the types of hardness of water ?
- 15. What is a basic buffer? Give an example ?
- 16. Write the structure of EDTA ?
- 17. What is the indicator used in this experiment?
- 18. What is hard water ?
- 19. Why is buffer added ?
- 20. What is total hardness of water ?

#### **2 ESTIMATION OF HCL BY CONDUCTOMETRIC TITRATION**

#### **INTRODUCTION:**

Conductometric titration is a laboratory method of quantitative analysis used to identify the concentration of a given analyte in a mixture. Conductometric titration involves the continuous addition of a reactant to a reaction mixture and the documentation of the corresponding change in the electrolytic conductivity of the reaction mixture. It can be noted that the electrical conductivity of an electrolytic solution is dependent on the number of free ions in the solution and the charge corresponding to each of these ions.

#### AIM:

To determine the strength of the strong acid by titration with strong base Conductometrically.

#### **APPARATUS:**

Conductivity Bridge, Conductivity cell, Burette, Beakers, Standard flask, pipette, Burette Stand e

#### **CHEMICALS REQUIRED:**

Sodium hydroxide, Hydrochloric acid

#### **PRINCIPLE:**

At first solution contain  $H^+$  and  $Cl^-$  ions. Since  $H^+$ ions posses greater mobility it follows that the conductivity is mainly due to  $H^+$  ions. The addition of NaOH is represented by the equation.

 $H^+ + Cl^- + Na^+ + OH^- \longrightarrow Na^+ + Cl^- + H_2O$ 

As NaOH is added the  $H^+$  ions are removed. The conductivity decreases as Na<sup>+</sup> ions do not process much mobility. As the neutralization point and solutions contains Na<sup>+</sup>ions and Cl<sup>-</sup> ions and will have minimum conductance value. If NaOH is further added this will add OH<sup>-</sup> ions and so the conductivity increases.

#### **PROCEDURE:**

A standard solution of 0.2N NaOH is prepared. Similarly 0.1N HCl is prepared. 20 ml of HCl is



taken in a 100 ml beaker and to it 20 ml of distilled water is added and kept in a thermostat. The conductivity cell is washed with distilled water and rinsed with acid soln. The cell is kept in acid containing beaker and it is connected to the bridge. The conductivity of the soln is measured by adjusting the reading. NaOH soln is taken into burette and add 1 ml of soln to acid, stirred well and conductance is measured. Each time 1 ml of base is added to acid stirred well and the conductance is measured. For every instance. Equal numbers of values are taken on either side of the point of maximum. Repeat the procedure of addition of 1 ml NaOH and noting the conductivity of the resulting solution. Take 20-25 readings .

S. No	Volume of NaOH	<b>Observed conductance</b>
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		

FORMULA:

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

N1 = Normality of NaOH = 0.1 N2 = ? V1 = Volume of Hcl = 20ml V2 = End point from the graph

#### **RESULT:**

The normality of strong acid (HCl) determined by titrating against a strong base (NaOH) =\_\_\_\_N

#### **VIVA QUESTIONS :**

- 1. Define conductance?
- 2. Give the definition of neutralization point?
- 3. What are the units of conductance?
- 4. Explain graph?
- 5. What is acid&Base?
- 6. What are conductors? How are they classified
- 7. Differentiate metallic and electrolytic conductors?
- 8. Define the specific conductance? With units.
- 9. Explain the term Equivalent conductance? With units.
- 10. Define Conductance? Write its units.
- 11. What is single electrode potential?
- 12. What is EMF of cell? How the emf of cell is calculated?
- 13. Calculate the molecular weight for Hcl

- 14. Differentiate Primary and Secondary cells?
- 15. Give the applications of Batteries?
- 16. Write cell reactions of Daniel cell?
- 17. Define electrochemical series? Write its applications?
- 18. Explain the construction of calomel electrode?
- 19. Mention the types of electrodes
- 20. Define a battery? How are they classified?

# **3 ESTIMATION OF Fe<sup>2+</sup> BY POTENTIOMETRY**

#### **INTRODUCTION:**

It is the procedure through which the quantity of the given test substance is determined by the measured addition of titrant until the entire test substance undergoes reaction. After the <u>titration</u> process, the potential difference between the two electrodes (namely the reference and indicator electrode) is measured in conditions where a thermodynamic equilibrium is maintained and the current passing through the electrodes does not disturb this equilibrium.

AIM: To estimate the Fe2+ ion by potentiometry

APPARATUS: Potentiometer, Beaker, Buirette, buirette stand .

CHEMICALS REQUIRED: Feso4 solution,KMno4 solution.

#### **PROCEDURE:**

potassium permanganate is used for determination of  $Fe^{2+}$ ,  $H_2O_2$  and oxalic acid. Potential at which the substance changes color must be such that the change occurs close to the equivalence point. permanganate has strong color by itself.

Approximately 0.1N Feso4 is prepared and standard decinormal solution of KMno4 is prepared. Exactly 20 ml of the acid is pipette out into a clean 100ml of beaker and a pinch of Quinhydrone is added which acts as indicator. Platinum electrode and calomel electrodes are dipped in the solution, and is being titrated is against KMno4 in burette. The solution is stirred well with a glass rod. The end reading is taken after adding definite amount of alkali. Finally after knowing the range in which the end point can be located, the whole experiment is repeatedly adding in steps of 1 ml in the end point.

S. No	Volume of KMnO4	Observed EMF
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		

### **CALCULATIONS:**

15	

#### **GRAPH:**

Two graphs are plotted of which one is between volume of alkali and observed emf and other is between volume of alkali and  $\Delta E/\Delta V$  Sigmoid curve



#### FORMULA:

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

N1 = Normality of KMnO4 = 0.1 N2 = ? V1 = Volume of Hcl = 20ml V2 = End point from the graph

#### **RESULT:**

The normality of strong acid determined by titrating against a strong base =\_\_\_\_\_N

#### **VIVA QUESTIONS:**

- 1. What is potentiometric titration?
- 2. What is the principle of potentiometric titration ?
- 3. What are the electrodes used in potentiometric titration?
- 4. What is neutralization point?
- 5. What is the color of feso4?
- 6.Write the different electrode reactions occur at the electrode?
- 7.Write the formula of ferrous ion ?
- 8. What are the advantages of potentiometric titration?
- 9.Define normality?
- 10. What is oxidation and reduction ?
- 11.Define std electrode potential?
- 12. What are the electrodes used in potentiometric titration?
- 13.Define indicator?
- 14. What are the chemicals used in potentiometry?
- 15. What is meant by reference electrode?
- 16. What is the color of Feso4?
- 17. What is the principle of potentiometric titration?
- 18. What is indicator electrode in potentiometry?
- 19. write the atomic number of manganese ?
- 20. What are the different types of potentiometers?

#### **4 ESTIMATION OF FERROUS IRON BY DICHROMETRY**

#### **INTRODUCTION:**

Dichrometry is the **most significant technique used in qualitative analysis in Chemistry**. It is a redox titration and involves the use of dichromates which are used to estimate the amount of analyze present in unknown chemical samples.

A dichromatic titration is **one in which potassium dichromate is used as an oxidizing agent**. When potassium dichromate is used in acidic aqueous media it is kept at a pH of 4.0 through the use of dilutions of sulfuric acid. Iodides and ferrous salts can be estimated with sodium dichromate solution.

**AIM:** To estimate the amount of ferrous iron present in the solution with the help of standard solution of Potassium Dichromate.

#### **APPARATUS REQUIRED:**

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

# **CHEMICALS REQUIRED:**

- 1. K 2 Cr 2 O 7
- 2. Diphenylamine(DPA)
- 3. Conc. H 2 SO 4
- 4. Ferrous ammonium sulphate(FAS)
- 5. 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.

$$K_2Cr_2O_7+4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3+4H_2O+3(O)$$
  
3[2FeSO<sub>4</sub>+H<sub>2</sub>SO<sub>4</sub>+ (O) → Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>+H<sub>2</sub>O]

 $K_2Cr_2O_7$ +6FeSO<sub>4</sub>+ 7H<sub>2</sub>SO<sub>4</sub> $\rightarrow$   $K_2SO_4$ + 3Fe<sub>2</sub> (SO<sub>4</sub>)<sub>3</sub>+  $Cr_2(SO_4)_3$ + 7H<sub>2</sub>O

### **PREPERATION OF CHEMICALS:**

**1. Preparation of standard Potassium Dichromate:** Weigh out accurately about 0.49gms of Potassium Dichromate into a 100 ml standard flask and dissolve the solid in a small quantity of distilled water. Make up the resulting solution with distilled water up to the mark and shake the flask well for uniform concentration.

**2. Preparation of acid mixture:** Mix up 100 ml of Phosphoric acid with 300ml of concentrated  $H_2SO_4$  in a reagent bottle and stopper it.

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

#### concentrated H 2 S0 4.

#### **PROCEDURE:**

 $\Box$  Rinse and fill the burette with standard K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution.

□ Pipette out 20ml of ferrous ammonium solution (FAS) into a 250ml conical flask and add 5ml of acid mixture and 2drops of diphenylamine indicator.

□ Titrate the solution against Potassium Dichromate taken in the burette till blue violet color is obtained as end point.

□ Repeat the titration to get concurrent values.

#### CALCULATIONS AND OBSERVATIONS:

S No.	Volume of Ferrous Solution (ml)	Burette reading		Volume of $K_2 Cr_2 O_7$ solution (ml)	
5.100	V <sub>2</sub>	Initial(ml)	Final(ml)	V <sub>1</sub>	
1					
2					
3					

### **CALCULATIONS:**

Normality of  $K_2Cr_2O_7 = N_1 = N$ 

Volume of  $K_2Cr_2O_7 = V_1 = ml$ 

Normality of Ferrous iron =N 2

Volume of Ferrous iron = V 2 ml

$$N_1 V_1 = N_2 V_2$$

Amount of iron (II) present in 100 ml of the given solution = =  $N_2X55.5$  =

10

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

**VIVA QUESTIONS:** 

1 Define dichrometry

2 What are the chemicals used in this experiment

3 Define oxidation reaction

4 What is the indicator used in Dichrometry estimation of iron?

5 What is the difference between ferric and ferrous?

6 What are the 4 types of titration?

7 Why K2Cr2O7 is strong oxidizing agent?

8 Why ferric is more stable than ferrous? (Ferric ion is more stable than ferrous ion. This is because **ferric ion has half filled d-subshell stable electronic configuration**.)

9 Write the the structure of diphenylamine

10 Write the structural formula of phosphoric acid

# 5(a) PREPARATION OF BAKELITE

# **INTRODUCTION:**

Bakelite is **a polymer made up of the monomers phenol and formaldehyde**. This phenol-formaldehyde resin is a thermosetting polymer.

**Polyoxybenzylmethylenglycolanhydride**, better known as **Bakelite**, is a <u>thermosetting phenol</u> formaldehyde resin, formed from a <u>condensation reaction</u> of <u>phenol</u> with <u>formaldehyde</u>. The first <u>plastic</u> made from synthetic components, it was developed by <u>Leo Baekeland</u> in <u>Yonkers</u>, <u>New York</u> in 1907

Because of its electrical <u>nonconductivity</u> and heat-resistant properties, it became a great commercial success. It was used in electrical insulators, radio and telephone casings, and such diverse products as kitchenware, jewelry, pipe stems, children's toys, and firearms.

AIM: - To prepare phenol formaldehyde resin. (Bakelite)

### CHEMICALS USED: -

Glacial acetic acid or 2N NaOH, 40% formaldehyde solution, Phenol, conc. H2SO4 /HCl

**APPARATUS REQUIRED**: -Glass rod, beakers, funnel, measuring cylinder, dropper and filter paper.

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

**PREPARATION:-** PFs are prepared by reaction of phenol with formaldehyde in the presence of acidic or

basic catalyst. The process may be carried out as follows

A mixture of phenol and formaldehyde are allowed to react in the presence of a catalyst. The process

involves formation of methylene bridges in *ortho*, *para* or *both ortho* and *para* positions. This results

first in the formation of linear polymer (Called *NOVALAC*) and then in to cross-linked polymer called phenol-formaldehyde resin or *Bakelite* 



(Cross-linked Polymer)

#### **PROCEDURE:** -

1. Place 5ml of glacial acetic acid and 2.5ml of 40% formaldehyde solution in a 500ml beaker and add 2 grams of phenol.

2. Add few ml of conc. Sulphuric acid into the mixture carefully. With in 5 min. a large mass of plastic is formed.

3. The residue obtained is washed several times with distilled water, and filtered product is dried and yield is calculated.

#### **USES:-**

They are used for making moulded articles such as radio and TV parts, combs, fountain pen barrels, phonograph records etc.

#### **PRECAUTIONS:**

1. The reaction is sometimes vigorous and it is better to be a few feet away from the beaker

while adding the  $H_2SO_4$  and until the reaction is complete.

2. The experiment should be preferably carried out in fume cupboard.

**RESULT:** - The weight of the phenol formaldehyde resin is \_\_\_\_\_\_g.

#### **VIVA QUESTIONS:**

- 1. Phenol formaldehyde is also called as.
- 2. What do you understand by resin?
- 3 Give main uses of the phenol formaldehyde resin.
- 4. What type of co-polymer is phenol formaldehyde resin?
- 5. Briefly describe the properties of phenolic resins
- 6. Write chemical equations for preparation of phenolic resins.
- 7. When was Bakelite first used?
- 8. What color is Bakelite?
- 9. Can Bakelite be recycled?
- 10. Is Bakelite A insulator?
- 11. Why is it called Bakelite?
- 12. Is Bakelite a natural polymer?
- 13. Why is Bakelite so good?
- 14. What are the disadvantages of Bakelite
- 15. Is Bakelite biodegradable?
  - 16 Is Bakelite a plastic?
  - 17. Why HCL is used in preparation of Bakelite?
  - 18. Does Bakelite melt on heating?
  - 19. What is the price of Bakelite?
  - 20. Is Bakelite used in cookware?

#### **5(b)PREPARATION OF THIOKOL RUBBER**

#### **INTRODUCTION:**

Thiokol is a trade mark for various organic polysulfide polymers. Thiokol rubber can be prepared by the condensation of 1, 2-dichloroethane with sodium polysulphide. Thiokol rubber is resistant to the action of oxygen, ozone and also to the action of petrol, lubricants and solvents. oil. Thiokol mixed with oxidizing agents can be used as a fuel in rocket engines.

#### **Definition of 'Thiokol'**

any of various synthetic rubbery materials resistant to oil, grease, and water, used as sealants, for hosing and tank linings, etc.

### AIM:

To synthesize Thiokol rubber using sodium polysulphide with 1, 2-Dichloroethane.

#### **APPARATUS:**

Beakers, glass rod, funnel etc.

#### **CHEMICALS REQUIRED:**

- 1. Sodium hydroxide
- 2. Powered sulphur
- 3. 1, 2-Dichloroethane
- 4. 5% H<sub>2</sub>SO<sub>4</sub>, etc

### **PRINCIPLE:** -

It is a rubbery white substance and is obtained by treating sodium polysulphide with 1, 2-Dichloroethane.

$$S_8 + 2 \text{ NaOH} \rightarrow \text{Na}_2 S_8$$

 $n(\text{Cl-CH}_2\text{-}\text{CH}_2\text{-}\text{Cl}) + n\text{Na}_2\text{S}_8 \rightarrow [\text{-}\text{CH}_2\text{-}\text{CH}_2\text{-}\text{S}\text{-}\text{S}\text{-}]_n + 2n\text{ NaCl}$ 

### **PROCEDURE:**

- 1. In a 100ml beaker dissolve 2gms NaOH in 50-60 ml of water.
- 2. Boil the solution and to this add in small lots with stirring 4 gms of powdered sulphur. During addition and stirring, the yellow solution turns deep red.
- 3. Cool it to 60-70 <sup>0</sup>C and add 10ml of 1, 2-Dichloroethane with stirring. Stir for an additional period of 20 min White rubber polymer separated out as lump.
- 4. Pour out the liquid from the beaker in the sink to obtain Thiokol rubber. Wash under the tap
- 5. Dry in the fold of filter paper, the yield is about 1.5 gms. Determine the solubility of the polymer in Benzene, Acetone, 5% H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> etc.

# **RESULT:**

Yield obtained = \_\_\_\_\_ gms

#### **VIVA QUESTIONS:**

- **1** What is Thiokol rubber?
- 2 What is property of Thiokol rubber?
- 3 What is the structure of Thiokol rubber?
- 4 What is the monomer of Thiokol?
- 5 Which type of polymer is Thikol rubber?
- 6 Is Thiokol synthetic rubber?
- 7 What are the uses of polysulfide rubber?
- 8 What are elastomers?
- 9 What is the chemical name of Thiokol rubber?
- 10 How is Thiokol produced?
- 11 What are the chemicals used for the preparation of Thiokol rubber
- 12 Define Thiokol
- 13 What are the apparatus used for the preparation
- 14 Write the uses of Thiokol rubber
- 15 Define condensation polymerization

# 6(a) Estimation of acid value of given lubricant oil

#### **INTRODUCTION:**

A lubricant (sometimes shortened to lube) is a substance that helps to reduce friction between surfaces in mutual contact, which ultimately reduces the heat generated when the surfaces move. It may also have the function of transmitting forces, transporting foreign particles, or heating or cooling the surfaces.

Lubrication is the process of reducing friction between touching surfaces moving relative to each other by introducing a lubricant between the surfaces, which is a material with a lower shear strength than the surfaces.

Lubricants do not necessarily completely prevent asperities, but they reduce their number and weaken their junctions. So lubrication also reduces the rate of sliding wear.

Good lubricants have high pour points (the lowest temperature at which an oil will flow), high viscosity indices (see later) and good resistance to oxidation.

#### AIM:

To determine the acid value of coconut oil

#### **APPARATUS:**

Burette, Beakers, pipette, Burette Stand

#### **CHEMICALS REQUIRED:**

Phenolphthalein indicator -Weigh 1 g of phenolphthalein and dissolve in 100 mL of ethanol. Sodium hydroxide titrant -Weigh accurately 4.0 g of sodium hydroxide and place it in a 1000-mL volumetric flask. Make up to the mark with water.

Ethanol-ether solution -Prepare a mixture of ethanol and diethyl ether (1:1, v/v). Neutralize with sodium hydroxide titrant and add 1.0 mL of phenolphthalein indicator until pink colouration is observed. Freshly prepare the solution.

#### **PRINCIPLE:**

In <u>chemistry</u>, **acid value** (or **neutralization number** or **acid number** or **acidity**) is the mass of <u>potassium hydroxide</u> (KOH) in <u>milligrams</u> that is required to neutralize one gram of <u>chemical</u> <u>substance</u>. The acid number is a measure of the number of <u>carboxylic acid</u> groups in a chemical compound, such as a <u>fatty acid</u>, or in a mixture of compounds. It is an important quality measurement of crude oil.

#### **PROCEDURE:**

weigh accurately a quantity of the fatty oil being examined as indicated in Table 1 and place it in a 250-mL conical flask, then add 50 mL of ethanol-ether solution. Shake it well. If necessary, reflux the mixture gently until the substance is completely dissolved.

Titrate the solution with sodium hydroxide / potassium hydroxide titrant until pink colouration can be observed which persists for 30 s.

Measure the volume of potassium hydroxide titrant used and calculate the acid value according to the following equation:

V Acid value = VKOH  $\times$  5.61 /W

Where,

VNaOH = Volume of sodium hydroxide titrant used (mL) W = Weight of the fatty oil being examined (g) [5.61 is the molecular weight of potassium hydroxide]

When the acid value is less than 10, it is suggested that a 10-mL semi-micro burette may be used for the titration.

#### **RESULT:**

The acid value of coconut oil is \_\_\_\_\_

#### **VIVA QUESTIONS:**

1. What is acid value ?

- 2. Write the formula of ethanol?
- 3. How can we calculate the acid value ?

4. What percentage of lauric acid in coconut oil?(50%, which makes it the most abundant natural source of lauric acid on earth)

5. What does an acid taste like?

6. How many carbons are there in lauric acid? (The 12-carbon **lauric acid** makes up about 50% of the **fatty acids** in coconut oil.)

- 7. What fatty acids are in coconut oil?
- 8. What is the IUPAC name of lauric acid?(dodecanoic acid)
- 9. Write the structure of lauric acid?



- 10.Write the formula of diethyl ether?
- 11. What are the chemicals used in this experiment ?
- 13. Write the common name of KOH?( caustic potash.)
- 14. What is the colour change at the end point ?
- 15. Name the indicator used in this experiment?

16. What is the structure of KOH? (Solid KOH is found in a rhombohedral crystalline structure)

17. What are the uses of lauric acid?(used in many soaps and shampoos in the form of sodium lauryl sulfate.)

- 18. What is a base number in a engine oil?
- 19. Define lubrication

20. What is the source of lauric acid?(it is abundant only in coconut)

# 6(b) Estimation of Viscosity of lubricant oil using Ostwald's Viscometer

# **INTRODUCTION:**

A lubricant (sometimes shortened to lube) is a substance that helps to reduce friction between surfaces in mutual contact, which ultimately reduces the heat generated when the surfaces move. It may also have the function of transmitting forces, transporting foreign particles, or heating or cooling the surfaces.

Lubrication is the process of reducing friction between touching surfaces moving relative to each other by introducing a lubricant between the surfaces, which is a material with a lower shear strength than the surfaces.

The most important property of an oil for lubricating purposes is its viscosity. Viscosity provides a measure of the resistance of a fluid to shearing flow.

Good lubricants have high pour points (the lowest temperature at which an oil will flow), high viscosity indices (see later) and good resistance to oxidation.

#### AIM:

To determine the absolute viscosity of a liquid by using Oswald's viscometer.

#### **APPARATUS:**

Oswald's viscometer, stop watch, density bottle, rubber bulbs, Beakers, etc.

#### **CHEMICALS REQUIRED:**

Standard liquid (water), test liquid etc.

#### PRINCIPLE: (POISEUILLE'S PRINCIPLE)

If a liquid flows with in a uniform velocity at a rate of 'V'in 't' seconds through a capillary tube of radius 'r' and length 1cm under a driving pressure 'p' dynes/  $cm^2$ . Then,

The co-efficient of viscosity is given as =

$$\eta = \prod r^4 t \Delta P / 8VL$$

- $\eta \implies$  Viscosity of liquid in poise
- $\Delta P \Longrightarrow$  pressure head i.e. dynes/ cm<sup>2</sup>.
- $r \Rightarrow$  radius of inner layer of capillary tube
- $L \implies$  length of capillary tube
- $V \implies$  volume of capillary tube
- $t \implies$  flow time in seconds

The poiseulles law is applicable only to linear flow or stream line flow. For a given Oswald's viscometer the length, radius and volume of liquids are constants and at end are combined to a single constant. The above equation can be written as

 $\eta = k t \Delta P$  In this eq.  $\Delta P$  depends on

- I. Density of liquid to be measured
- II. Acceleration due to gravity
- III. the difference due to gravity is constant Then

The viscosity of liquid may be expressed as

 $\eta_1 = kt_1 \rho_1$  (viscosity Standard liquid (water))

 $\eta_2 = kt_2 \rho_2$ (viscosity of test liquid)

Relative viscosity  $\Rightarrow \eta_1/\eta_2 = t_1 \rho_1/t_2 \rho_2$ 

Units: (CGS)  $\Rightarrow$  dynes-sec/cm<sup>2</sup> or poise

#### **PROCEDURE:**

Clean thoroughly and dry the Oswald's viscometer, a definite volume of standard liquid is allow to flow into 'A ' arm such that it raises above the values X and Y. The same procedure is repeated with the test liquid and note the time by stop clock

#### **CALCULATIONS:**

S. No	Standard liquid (t <sub>1</sub> )	Test liquid (t <sub>2</sub> )	
TRIAL-I			
TRIAL-II			
TRIAL-III			
Weight of en Weight of en Weight of en Density of w	npty density bottle (W <sub>1</sub> ) = npty bottle + water (W <sub>2</sub> ) = npty bottle + liquid (W <sub>3</sub> ) = rater ( $\rho_{1}$ ) = $\frac{W2 \cdot W1}{25}$ = quid ( $\rho_{2}$ ) = $\frac{W3 \cdot W1}{25}$ =	gm gm gm	

Viscosity Standard liquid ( $\eta_1$ ) at 25°C= 1.0019cps

$$\eta_2 = t_2 \rho_2 / t_1 \rho_1 \times \eta_1$$

#### **RESULT:**

Absolute viscosity of a given liquid ( $\eta_2$ ) = \_\_\_\_\_ cps

#### **VIVA QUESTIONS:**

- 1. Define viscocity?
- 2.Write the units of viscocity?
- 3. What are the apparatus are used to determine the viscocity?

4.Discuss about lubricant?
5.What is formula of viscocity?
6.Define flash point of a lubricant?
7. Classification of lubricant
8.Define cloud and pour point of a lubricant?
9.What is density?
10. Name the units of density?
11.What are the chemicals are used in this experiment?
12. Is glycerol a lubricant?
13. Write the IUPAC name of glycerol
14. What are the apparatus are used in determination of flash point?
15.What is lubrication?
16. Write the structure of glycerol
17. What is the volume of density bottle
18.Write the examples of vegetable oils?
19.What are refractories?

# 7 Preparation of Hand sanitizer(Iso propyl alcohol)

# **INTRODUCTION:**

20. Define lubrication

**Hand sanitizer** (also known as **hand antiseptic**, **hand disinfectant**, **hand rub**, or **handrub**) is a liquid, <u>gel</u> or foam generally used to kill many <u>viruses/bacteria/microorganisms</u> on the <u>hands</u>. In most settings, <u>hand washing</u> with <u>soap</u> and water is generally preferred.

Alcohol based hand sanitizer is recommended by the United States <u>Centers for Disease Control</u> and <u>Prevention</u> (CDC), but only if soap and water are not available. The CDC recommends the following steps when using an alcohol-based hand sanitizer:

- 1. Apply product to the palm of one hand.
- 2. Rub hands together.
- 3. Rub the product over all surfaces of hands and fingers until hands are dry.
- 4. Do not go near flame or gas burner or any burning object during application of hand sanitizer.

Alcohol-based hand sanitizer works against a wide variety of <u>microorganisms</u> but not <u>spores</u>. Compounds such as <u>glycerol</u> may be added to prevent drying of the skin. Some versions contain fragrances; however, these are discouraged due to the risk of allergic reactions.

hand sanitizers can be classified as one of two types: <u>alcohol</u>-based or alcohol-free.

Alcohol-based products typically contain between 60 and 95 percent alcohol, usually in the form of <u>ethanol</u>, <u>isopropanol</u>, or *n*-propanol. At those concentrations, alcohol immediately denatures <u>proteins</u>, effectively neutralizing certain types of microorganisms. Isopropanol or isopropyl alcohol is a clear, colorless liquid that is a major component of rubbing alcohol as well as regular household items such as cleaners, disinfectants, and hand sanitizers; it also can be found in pharmaceuticals.

Alcohol-free products are generally based on <u>disinfectants</u>, such as benzalkonium chloride (BAC), or on <u>antimicrobial agents</u>, such as triclosan.<sup>1,6,7</sup> The activity of disinfectants and antimicrobial agents is both immediate and <u>persistent</u>.

#### AIM:

To prepare iso propyl alcohol based hand sanitizer

#### **APPARATUS:**

Measuring jar, Beaker, Conical flask

#### **CHEMICALS REQUIRED:**

isopropyl alcohol or ethanol, hydrogen peroxide, glycerol, sterile distilled or boiled cold water

#### **PROCEDURE:**

Mix 450 mL 99% isopropyl alcohol, 9 mL glycerin, 16 mL cold drinking water, and 25 mL 3% hydrogen peroxide in a beaker.

Mix the resulting solution well with a glass rod to ensure that the alcohol is evenly distributed throughout the gel, and let sit for 72 hours in a cool, dark place.

Distribute the sanitizer into convenient containers with dispensers.

#### **VIVA QUESTIONS:**

**1** What is isopropyl alcohol?

2 How do you make hand sanitizer from isopropyl alcohol?

3 What is the process of making hand sanitizer?

4 Define sanitizer ?(a substance or product that is used to reduce or eliminate pathogenic agents (such as bacteria) on surfaces.)

- 5 Can you use isopropyl alcohol as hand sanitizer?
- 6 What is the chemical formula for isopropyl alcohol?(C3H8O)
- 7 What is the common name of isopropyl alcohol?
- 8 Write uses of iso propyl alcohol
- 9 What is the use of glycerol in sanitizer
- 10 What is the function of hydrogen peroxide in hand sanitizer?

It **kills bacteria** that can get into the bottles or the sanitizer as you make it. Take extra care with this step, since hydrogen peroxide may irritate your skin.

# **8 Virtual lab experiments:**

# (a) Construction of Fuel cell and its working

# **INTRODUCTION:**

A fuel cell can be defined as an electrochemical cell that generates electrical energy from fuel via an electrochemical reaction.

Fuel cells require a continuous input of fuel and an <u>oxidizing agent</u> (generally oxygen) in order to sustain the reactions that generate the electricity. Therefore, these cells can constantly generate electricity until the supply of fuel and oxygen is cut off.

A fuel cell is similar to <u>electrochemical cells</u>, which consists of a cathode, an anode, and an electrolyte. In these cells, the electrolyte enables the movement of the protons.

Today, these devices are used as the primary or secondary source of power for many facilities including industries, commercial buildings, and residential buildings.

# Working of Fuel Cell:

The working of this fuel cell involved the passing of hydrogen and oxygen into a concentrated solution of sodium hydroxide via carbon electrodes. The cell reaction can be written as follows:

Cathode Reaction:  $O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$ 

Anode Reaction:  $2H_2 + 4OH^- \rightarrow 4H_2O + 4e^-$ 

Net Cell Reaction:  $2H_2 + O_2 \rightarrow 2H_2O$ 

However, the reaction rate of this electrochemical reaction is quite low. This issue is overcome with the help of a catalyst such as platinum or palladium. In order to increase the effective surface area, the catalyst is finely divided before being incorporated into the electrodes.



# **Applications of fuel cell**

Fuel cell technology has a wide range of applications. Currently, heavy research is being conducted in order to manufacture a cost-efficient automobile which is powered by a fuel cell. A few applications of this technology are listed below.

- Fuel cell electric vehicles, or FCEVs, use clean fuels and are therefore more eco-friendly than internal combustion engine-based vehicles.
- They have been used to power many space expeditions including the Appolo space program.
- Generally, the byproducts produced from these cells are heat and water.
- The portability of some fuel cells is extremely useful in some military applications.
- These electrochemical cells can also be used to power several electronic devices.
- Fuel cells are also used as primary or backup sources of electricity in many remote areas.

# (b) Smart materials for Biomedical applications

# **INTRODUCTION:**

Smart materials are having ability to change its shape according to the external stimulus like

temperature, pressure, electric field, magnetic field, etc. Because of their responsiveness, smart materials are also known as responsive materials.

These are usually translated as "active" materials although it would be more accurate to say "reactive" materials.

The materials used for biomedical applications are:

**Metals, ceramics, plastic, glass, and even living cells and tissue** all can be used in creating a biomaterial. They can be reengineered into molded or machined parts, coatings, fibers, films, foams, and fabrics for use in biomedical products and devices.

Smart materials includes **piezoelectric materials, magnetorheostatic materials**, electrorheostatic materials, and shape memory alloy.

# **TYPES OF SMART MATERIALS**

#### A Shape Memory Alloys (SMAs)

Shape memory alloys (SMA) are materials that "remember" their original shape and can go back to this original shape after deformation under a stimulus. They are also known as smart alloys or memory metals.

### **B** Piezoelectric Materials

The term piezoelectricity is a blend of two terms: "piezo" which is a Greek term meaning pressure and "electricity" referring to electric charges. By the application of stress or strain piezoelectric material changes the mechanical energy into electrical energy and vice-versa. Similarly, piezoelectric actuators convert electrical signals into a mechanical movement which is used for adjusting mirrors, lenses and various automotive parts.

### C. Magneto-Rheological Fluids

Magneto-Rheological Fluids (MRFs) will change their rheological properties like stress and viscosity on the application of the magnetic field. Magneto-Rheological Fluids (MRFs) are also called Magneto-Sensitive Smart Materials. Magneto-Rheological Fluids (MRFs) have the properties such as visco-elastic in nature, magnetic property, light in weight, controllable modulus and excellent sound absorbing.

### D. Electro-Rheological Fluids

The Electro-Rheological Fluids (ERFs) is the suspension of very small particles in electrical insulating fluid when the electric field is applied, they will rapidly form a solid-like structure in the direction of the field. Electro-Rheological Fluids (MRFs) have the properties such as stiff, damping coefficient is changed in the electric field, high dielectric constant, interfacial bond strength, constable rheology and dielectric in nature.

# E. Optical Fiber

A flexible and transparent fiber which is made by drawing glass/ plastic to a diameter slightly thicker than the diameter of the human hair is called Optical Fiber. These are used quite often to transmit the light between the ends of the fiber.

# **APPLICATIONS OF SMART MATERIALS:**

SMAs have been used in bioengineering applications such as dental wires such as those used in dental braces, mending broken bones using metal plates, and for medical devices that help open clogged veins and arteries. They are used as wires and tubes in applications with hot fluids flowing through them. These materials are ideal as they can retain their shape even in a heated environment.

Piezoelectric materials can be employed in **monitoring many bodily signals** because they convert mechanical energy into an electrical signal. They are especially applicable to monitoring dynamic pressure changes; many human vital signs consist of rhythmic activities like the heartbeat or breathing.

Magnetorheological (MR) fluid, whose rheological properties can be changed reversibly by applied magnetic field, offers superior capabilities and opportunities since its invention. The most crucial feature of MR fluid is its controllable and continuous yield stress. Taking this advantage, MR fluid is gaining popularity in various medical applications to meet their force/torque requirements. With MR fluid, natural and stable limb motions in lower limb prostheses, exoskeletons, and orthoses, flexible muscle trainings in rehabilitation devices, and high transparency and resolution haptic feedback can be realized.

Fiber optic technology allows surgeons to repair organs, diagnose joint problems, and remove diseased tissues, leaving the patient with a shorter recovery time than more invasive surgical methods.