

**ENGINEERING CHEMISTRY**  
(Common to CIVIL & MECHANICAL Engineering)  
**ENGINEERING CHEMISTRY LAB**  
(LABORATORY MANUAL)

**LAB CODE: R23BS04**

**SCHEME: R23**



**DEPARTMENT OF BASIC SCIENCES AND HUMANITIES**

**BEHARA COLLEGE OF ENGINEERING & TECHNOLOGY**

Approved by AICTE, New Delhi, Affiliated to JNT University, Gurajada Vizianagaram

88<sup>th</sup> ward , Narava(V), Visakhapatnam, 530027



## VISION

To establish a strong foundation in basic sciences such as Mathematics, Physics, Chemistry, Humanities with good communication skills, discipline, and ethics for lifelong learning for the students of different engineering disciplines to apply in relevant Engineering practice and Inter- disciplinary research to attain their professional and academic goals.

## MISSION

- To cultivate in students, the needed Life Skills and to strengthen fundamentals of Basic sciences, Humanities and Environmental Sciences as to enable them succeed in the highly competitive world.
- To impart academic and life skills with high degree of social consciousness and human values
- To promote co-curricular and extra-curricular activities for overall personality development of students.
- To be a catalyst for holistic education, shaping future engineers to be intellectually curious, socially responsible, and adaptable in a technologically evolving world.



By the completion of engineering program, the students will be to:

**PROGRAM EDUCATIONAL OBJECTIVES (PEOS):**

**PEO1:**

Learn Skills, skill to understand, analyse, Verify the fundamental concepts with experiments.

**PEO2:**

Problem solving using hardware and software tools & Lifelong learning, Capability to pursue career in industry or higher studies with continuous learning. (Problem-Solving Skills).

**PEO3:**

Entrepreneurship Skills, Leadership qualities, team spirit, multi-disciplinary approach, character moulding, effective communication skills, lifelong learning and sense of responsibility towards society for a successful professional career. (Professional Career).

**PROGRAM SPECIFIC OUTCOMES (PEOS):**

**PSO-1:** Determine the cell constant and conductance of solutions.

**PSO-2:** Prepare advanced polymer materials.

**PSO-3:** Determine the physical properties like surface tension, adsorption and viscosity.

**PSO-4:** Estimate the Iron and Calcium in cement.

**PSO-5:** Calculate the hardness of water.



## **PROGRAM OUTCOMES (POs)**

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.



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## SYLLABUS

### ENGINEERING CHEMISTRY LAB

#### Course Objectives:

- Verify the fundamental concepts with experiments.

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

**CO1:** Determine the cell constant and conductance of solutions.

**CO2:** Prepare advanced polymer materials.

**CO3:** Determine the physical properties like surface tension, adsorption and viscosity.

**CO4:** Estimate the Iron and Calcium in cement.

**CO5:** Calculate the hardness of water.

#### List of Experiments:

1. Determination of Hardness of a groundwater sample.
2. Estimation of Dissolved Oxygen by Winkler's method
3. Determination of Strength of an acid in Pb-Acid battery
4. Preparation of a polymer (Bakelite)
5. Determination of percentage of Iron in Cement sample by colorimetry
6. Estimation of Calcium in port land Cement
7. Preparation of nanomaterials by precipitation method.
8. Adsorption of acetic acid by charcoal
9. Determination of percentage Moisture content in a coal sample
10. Determination of Viscosity of lubricating oil by Redwood Viscometer 1
11. Determination of Viscosity of lubricating oil by Redwood Viscometer 2
12. Determination of Calorific value of gases by Junker's gas Calorimeter

#### Reference:

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





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## During the lab class

### DO'S

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. Use protective clothing all the time (e.g. lab coat and safety glasses).
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. Wash acid, base, or any chemical spill off of yourself immediately with large amounts of water.

Notify your teacher of the spill.

4. 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.

5. 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.

6. Place the reagents in a systemic manner.

7. If you burn yourself on a hot object, immediately hold the burned area under cold water for 15 minutes. Inform your teacher.

8. 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



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book bags should not be on the lab table. Passageways need to be clear at all times.

9. Always add acid to water and stir the solution while adding the acid. Never add water to an acid.
10. Report all accidents to your teacher.
11. Thoroughly clean your laboratory work space at the end of the laboratory session. Make sure that all equipments are clean, and returned to its original place.

### DON'T

1. 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.
2. Wear bulky or dangling clothing.
3. Eat or drink in the laboratory. Don't chew on the end of a pen which was lying on the lab bench.
4. Use Mobile Phones.
5. Directly touch any chemical with your hands. Never taste materials in the laboratory.
6. Waste the reagents.
7. 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.
8. When weighing never place chemicals directly on the balance pan. Never weigh a hot object.
9. 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.
10. Absolutely no running, practical jokes, or horseplay is allowed in the laboratory.
11. Allow the reagent bottles to accumulate on the bench.



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## **ABOUT THE LAB**

**In this lab students will be able to study and Students will analyse different water samples collected from their residential areas and from other places and the results obtained to be compared with Indian standards, based on the position of the metals in the electrochemical series a model electrochemical cell is constructed and the potential values are determined and effect of metal ion concentration is studied.**

## **SIGNIFICANCE OF THE LAB**

**Engineering Chemistry laboratory is used for the methods for estimation of total hardness by EDTA method, Total dissolved salts, dissolved oxygen, Bacterial contents etc present in water and also used for the estimation of viscosity and calorific value by using instrumental based experiments.**





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|                               |   |
|-------------------------------|---|
| <b>Course Designed by</b>     | Department of Basic sciences and Humanities   |
| <b>Category</b>               | <ul style="list-style-type: none"><li>• Quantitative analysis Experiments</li></ul> |
|                               | <ul style="list-style-type: none"><li>• Instrumentation based Experiments</li></ul> |
| <b>Broad Area of Syllabus</b> | Engineering CHEMISTRY LABORATORY  |



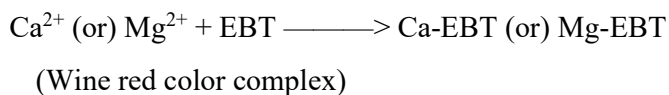
## Experiment-1 DETERMINATION OF HARDNESS OF GORUND WATER

**AIM:** To estimate hardness of ground water by using standard solution of EDTA.

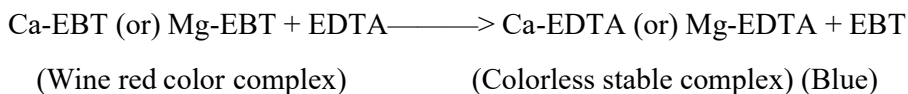
**APPARATUS:** Burette, Pipette, Conical flask, Beakers, Standard flask, Burette stand and funnel etc.

**CHEMICALS REQUIRED:** Buffer solution, EDTA solution, Eriochrome black-T, Ground water etc.

**PRINCIPLE:** Hard water which contains  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  ions which forms wine red color complex with the indicator.



EDTA forms a color less complex with the metal ions ( $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ )



When free ions are not available, EDTA extracts the metal from (ion) metal ion indicator complex, there by releasing the free indicator.

### PROCEDURE:

#### STANDARDISATION OF EDTA:

About x gms of potassium EDTA sample is taken in clean dry weighing bottle and its weight is found correctly by using physical balance. Transfer the substance in to 100ml volumetric flask using funnel. Dissolve the substance in minimum quantity of distilled water. Make up the solution upto the mark with distilled water and shake the flask for uniform concentration. Find out the weight of the empty weighing bottle. The difference between two weights gives the weight of the substance. Then calculate normality of the EDTA.



**OBSERVATIONS AND CALCULATIONS:**

Weight of the weighing bottle+Substance (W1)=                      gms

Weight of the empty weighing bottle (W2)=                      gms

Weight of the substance= (W1-W2)= \_\_\_\_\_ gms

$$\text{Normality of EDTA (N1)} = \frac{\text{Weight of substance X100}}{\text{Equivalent EDTA X volume of solution (100ml)}} = \text{M}$$

**ESTIMATION OF HARDNESS OF GROUND WATER:** The burette is filled with standard EDTA and initial reading is noted. Pipette is cleaned and rinse with given sample solution and 100 ml of the given ground water is pipette out in a clean conical flask and add 2ml of ammonia solution followed by few drops of Eriochrome black-T indicator. It is titrated by adding EDTA from burette till blue color is obtained. The final reading is noted, difference between final and initial volumes gives the volume of the EDTA solution required to react with 100 ml of given ground water sample. The experiment is repeated to get two consecutive readings coinciding. The normality of the given solution is calculated.

| S.No. | VOLUME OF CALCIUM SOLUTION | BURETTE READINGS(ml) |       | VOLUME OF EDTA |
|-------|----------------------------|----------------------|-------|----------------|
|       |                            | Initial              | Final |                |
| 1.    | 20 ml                      | 0                    |       |                |
| 2.    | 20ml                       |                      |       |                |
| 3.    | 20 ml                      |                      |       |                |

**Calculations:**

Volume of EDTA solution (V<sub>1</sub>) =                      ml

Molarity of EDTA solution (M<sub>1</sub>) =

Volume of water solution (V<sub>2</sub>) =                      20 ml

molarity of water solution (M<sub>2</sub>) =                      =

Hardness of given ground water sample= **N<sub>2</sub>X100X1000 ppm**



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### RESULT

The hardness of ground water sample is \_\_\_\_\_ mg/L



## EXPERIMENT-2

### PH metric titration of (i) Strong acid vs. Strong base (ii) Weak acid vs. Strong base.

#### (i) Strong acid VS Strong base

**AIM:** To determine the strength of the strong acid by titration with strong base by using conduct meter.

**APPARATUS:** Conductivity Bridge, Conductivity cell, Burette, Beakers, Standard flask, pipette, Burette Stand etc.

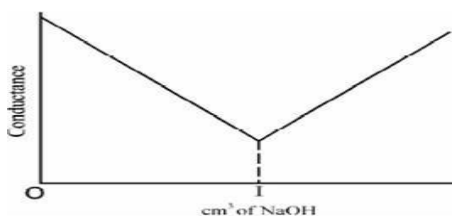
**CHEMICALS REQUIRED:** Sodium hydroxide, Hydrochloric acid

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



As NaOH is added the  $H^+$  ions are removed. The conductivity decreases as  $Na^+$  ions do not possess much mobility. As the neutralization point is reached the solution contains  $Na^+$  ions and  $Cl^-$  ions and will have minimum conductance value. If NaOH is further added this will add  $OH^-$  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 solution is measured by adjusting the reading. NaOH solution is taken into burette and adds 1 ml of solution 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.







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### Tabular form and calculations:

| S. No | Volume of NaOH | Observed conductance |
|-------|----------------|----------------------|
| 1     |                |                      |
| 2     |                |                      |
| 3     |                |                      |
| 4     |                |                      |
| 5     |                |                      |
| 6     |                |                      |
| 7     |                |                      |
| 8     |                |                      |
| 9     |                |                      |
| 10    |                |                      |
| 11    |                |                      |
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| 16    |                |                      |
| 17    |                |                      |
| 18    |                |                      |
| 19    |                |                      |
| 20    |                |                      |
| 21    |                |                      |
| 22    |                |                      |
| 23    |                |                      |
| 24    |                |                      |
| 25    |                |                      |



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**FORMULA:**  $N_1V_1 = N_2V_2$

**RESULT:** The normality of strong acid (HCl) determined by  
Titrating against a strong base (NaOH) = \_\_\_\_\_ .N



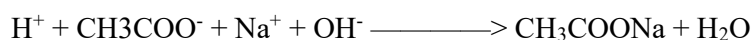
## (ii) Weak acid VS Strong base

**AIM:** To determine the strength of the weak acid by titration with strong base by using conduct meter.

**APPARATUS:** Conductivity Bridge, Conductivity cell, Burette, Beakers, Standard flask, pipette, Burette Stand etc.

**CHEMICALS REQUIRED:** Sodium hydroxide,

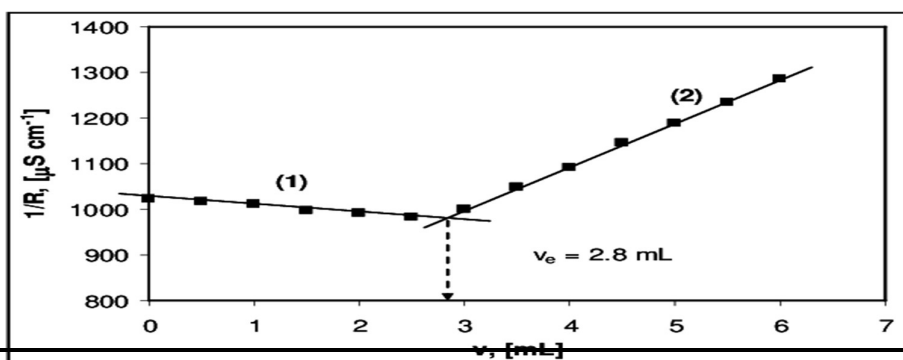
**PRINCIPLE:** At first solution contain  $\text{CH}_3\text{COO}^-$  and  $\text{H}^+$  ions. Since  $\text{H}^+$  ions possess greater mobility it follows that the conductivity is mainly due to  $\text{H}^+$  ions. The addition of  $\text{NaOH}$  is represented by the equation.



As  $\text{NaOH}$  is added the  $\text{H}^+$  ions are removed. The conductivity decreases as  $\text{Na}^+$  ions do not possess much mobility. As the neutralization point is reached the solution contains  $\text{Na}^+$  ions and  $\text{Cl}^-$  ions and will have minimum conductance value. If  $\text{NaOH}$  is further added this will add  $\text{OH}^-$  ions and so the conductivity increases.

**PROCEDURE:** A standard solution of 0.2N  $\text{NaOH}$  is prepared. Similarly 0.1N  $\text{CH}_3\text{COOH}$  is prepared. 20 ml  $\text{CH}_3\text{COOH}$  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 solution is measured by adjusting the reading.  $\text{NaOH}$  solution is taken into burette and adds 1 ml of solution 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  $\text{NaOH}$  and noting the conductivity of the resulting solution. Take 20-25 readings.

**Graph :**





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### Tabular form and calculations:

| S. No | Volume of NaOH | Observed conductance |
|-------|----------------|----------------------|
| 1     |                |                      |
| 2     |                |                      |
| 3     |                |                      |
| 4     |                |                      |
| 5     |                |                      |
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**FORMULA:**  $N_1V_1 = N_2V_2$

**RESULT:** The normality of weak ( $\text{CH}_3\text{COOH}$ ) determined by titrating against a strong base (NaOH) =





## EXPERIMENT -3

### Determination of cell constant and conductance of solutions

**Aim:** - Determination of cell constant of a conductivity cell.

**Apparatus:** - Conduct meter, Conductivity cell, beaker, Standard flask etc.

**Chemicals:** - Potassium chloride, distilled water etc.

**Theory:** - Conductance is the reciprocal of resistance. It is depend upon three factors, number of ions, and nature of ions and mobility of ions towards their respective electrodes. The specific conductance of electrolyte is decreased by increasing its dilution and equivalent conductance is increased by increasing its dilution. The observed conductivity of an electrolyte will be equal to its specific conductivity if cell constant is one.

**Procedure:** - Connect conductivity cell to Conductivity Bridge and keep the cell deepen in distilled water. Satisfactory and reproducible results demand the utmost care and cleanliness in the preparation of solutions and their transfer. Glassware should be cleaned thoroughly with cleansing acid, rinsed repeatedly with tap water, distilled water and finally with conductance water. Do not use organic solvents for drying, use a drying oven. The electrodes of the conductance cell must be immersed in conductance water whenever the cell is not in use. Never touch the electrodes. All solutions should be prepared using conductance water. In making conductivity measurements, allow the cell to remain in the constant temperature bath for at least fifteen minutes before balancing the bridge for the final reading.

Prepare the solutions of 0.1, 0.01, 0.02, 0.005 and 0.001 N of KCl.

#### **PART I – Determination of cell constant.**

Note down the conductance of 0.1 N and 0.02 N KCl solutions by using conductometer and Determine the cell constant.

#### **PART II –**

Determine the conductance of all prepared KCl solutions and calculate the specific and equivalent conductance of each solution. Plot a graph Equivalent conductance against concentration of solution.

Formula:

Cell constant = Specific conductance/ observed conductivity

Specific conductance = Cell constant x observed conductivity

Equivalent conductance =  $K \times 1000/C$



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### Part-1

**Table: 1**

**Cell constant:**

| S. No | Conc. OF KCl solution | Observed Conductance | Specific (k)Conductance | Cell Constant | Mean |
|-------|-----------------------|----------------------|-------------------------|---------------|------|
| 1     | 0.1                   |                      |                         | 0.970         |      |
| 2     | 0.02N                 |                      |                         | 0.970         |      |

### Part-II

**Equivalent conductance and specific conductance**

| S. No | Conc. of KCl solution | Observed Conductance | Specific Conductance (k) | Equivalent conductance ( $\lambda$ ) |  |
|-------|-----------------------|----------------------|--------------------------|--------------------------------------|--|
|       |                       |                      |                          |                                      |  |
|       |                       |                      |                          |                                      |  |
|       |                       |                      |                          |                                      |  |
|       |                       |                      |                          |                                      |  |

**Calculations:-**

1. Specific conductance:-

2. Equivalent Conductance:-

**Result:** - The cell constant of the given conductivity cell is = ...../cm



## EXPERIMENT-4

### Potentiometer - determination of redox potentials and emfs

#### INTRODUCTION

Experiment you are going to perform a redox titration. EMF change during the titration is measured by potentiometer. The equivalence point of the titration is detected by plotting a titration curve similar to the previous experiment.

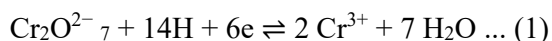
#### Objectives

After performing this experiment you will be able to:

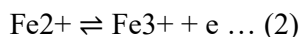
- Discuss how the potential changes with relative concentration of oxidised/reduced form,
- perform a redox titration of ammonium iron (II) sulphate using potassium dichromate as oxidizing agent,
- determine the equivalence point of the redox titration by plotting titration curve using potential change values and amount of oxidizing agent added during titration,
- estimate the strength of iron (II) ions in the given solution,
- state the advantages of potentiometry in redox titrations, and
- practice the precautions while performing a potentiometric titration experiment.

#### 4.2 PRINCIPLE

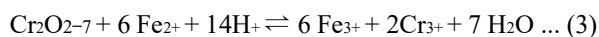
This is an example of redox titration and is based on the oxidation-reduction reaction between the titrand and the titrant. Here the end point is detected using a potentiometer. From the Nernst equation, you know that the potential of a given reaction will depend on the relative concentration of oxidised/reduced form. During their titration, the solution potential changes due to the change in the concentration of oxidised/reduced form. At one stage, where either of the form is absent i.e. at the end point, there is a sharp change in potential. Potassium dichromate is an oxidising agent and in acid medium; it follows the half reaction to give Cr (III) as the reduction product.



While  $\text{Fe}^{2+}$  which is used to titrate  $\text{K}_2\text{Cr}_2\text{O}_7$  gets oxidised to  $\text{Fe}^{3+}$  as per the reaction



The overall ionic equation of this titration can be obtained by adding the above two:





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### REQUIREMENTS:-

#### Apparatus:-

Burette, pipette, volumetric flasks, beakers, magnetic stirrer, potentiometer, SCE, platinum indicator electrode, connecting wires etc.

#### Chemicals:-

Potassium dichromate, ammonium iron (II) sulphate and sulphuric acid

### PROCEDURE

1. Using 100 cm<sup>3</sup> volumetric flasks prepare of 0.02 M potassium dichromate solution and 0.10 M ammonium iron (II) sulphate solution. You may have to add sufficient amount of dilute acid to prepare ammonium iron (II) sulphate solution.
2. Take 25 cm<sup>3</sup> of given Fe<sup>2+</sup> solution and add 25 cm<sup>3</sup> dilute H<sub>2</sub>SO<sub>4</sub> acid and 50 cm<sup>3</sup> of distilled water in a 250 cm<sup>3</sup> beaker.
3. SCE is used as the Reference electrode. Platinum metal foil, dipped in Fe<sup>2+</sup> solution is used as the indicator electrode.
4. Carry out necessary connections as shown in Fig. 3.1.
5. Standardise the potentiometer using a standard cell before replacing it with the working cell.
6. Add 2 cm<sup>3</sup> of 0.02 M K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution from burette, operate the magnetic stirrer for 2 minutes, stop it, wait it for 1 minute and measure the E.M.F.
7. Repeat the above step, each time adding two more cm<sup>3</sup> of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> at a time and go on noting the E.M.F. after each addition.
8. When the volume reached near about 1 cm<sup>3</sup> of the expected equivalence point (approximate), add the solution from burette in 0.5 cm<sup>3</sup> instalments and note the potential each time.
9. Continue adding these instalments even after the equivalence point (This can be easily observed from the change in measured potential). The change becomes very small. Continue for another 5-6 additions. Note the potential readings.
10. Record the observations in the Observation Table 1. Follow the same procedure for plotting the graphs and locate the equivalence point as given in Experiment No. 3.







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### CALCULATIONS:-

From the plotted curve, the volume of  $K_2Cr_2O_7$  used corresponding to the equivalence point =  $V \text{ cm}^3$  .....

Apply the molarity equation to calculate molarity of potassium dichromate solution.

$$M_1V_1 = 6M_2V_2$$

Ferrous ammonium Sulphate  $K_2Cr_2O_7$

$$M_2 = \frac{M_1V_1}{6V_2} \text{ mol dm}^{-3}$$

.....  $\text{mol dm}^{-3}$

### RESULT:-

Strength of the potassium dichromate solution is -----



### EXPERIMENT-5

## Determination of Strength of an acid in Pb-Acid battery

**Aim:** To determine the strength of acid in Lead-Acid battery

**Apparatus:** Burette, Conical flask, Beaker, Burette stand, Pipette and Wash bottles

**Chemicals required:** Acid from Lead acid battery, NaOH, Oxalic acid, Phenolphthalein indicator

#### Principle:

Strength of acid in Lead-Acid battery, measure of its ability to neutralize with bases to resist change of pH value of acid due to presence of mineral acids like  $H_2SO_4$ .



#### Procedure:

##### Standardization of Sodium Hydroxide:

Take 20 ml of oxalic acid solution in to the conical flask and add 2 to 3 drops of Phenolphthalein indicator, resulting the complete solution is colorless. The solution is titrated against with Sodium hydroxide solution. The sample solution changes colorless to pink color. The titration stops and note down the burette value. Then titration should be repeated until getting concrete readings.

##### Determination of strength of an acid:

Take 20 ml of acid solution from lead acid battery in to the conical flask and add 2 to 3 drops of Phenolphthalein indicator, the complete solution is colorless. The solution is titrated against with sodium hydroxide solution. The sample solution changes colorless to pink color. The titration stops and note down the burette value. Then titration should be repeated until getting concrete readings.



**Standardization of Sodium hydroxide:**

| S. No | Volume of sample solution | Burette reading |       | Volume of oxalic acid |
|-------|---------------------------|-----------------|-------|-----------------------|
|       |                           | Initial         | Final |                       |
|       |                           |                 |       |                       |
|       |                           |                 |       |                       |
|       |                           |                 |       |                       |

**Calculations:**

$$N_1V_1 = N_2V_2$$

Concentration of Sodium Hydroxide ( $N_1$ ) = ?

Volume of sodium hydroxide ( $V_1$ ) =            ml

Concentration of Oxalic acid ( $N_2$ ) =            N

Volume of Oxalic Acid ( $V_2$ ) =            ml

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

Concentration of Sodium hydroxide solution  $N_1$  =

**1. Strength of acid in Lead-Acid battery**

| S. No | Volume of sample solution | Burette reading |       | Volume of Sodium Hydroxide |
|-------|---------------------------|-----------------|-------|----------------------------|
|       |                           | Initial         | Final |                            |
|       |                           |                 |       |                            |
|       |                           |                 |       |                            |
|       |                           |                 |       |                            |

**Calculations:**

$$N_1V_1 = N_2V_2$$

Concentration of Sodium Hydroxide ( $N_1$ ) =

Volume of sodium hydroxide ( $V_1$ ) =            ml

Concentration of Sulphuric acid ( $N_2$ ) = ?

Volume of Sulphuric acid ( $V_2$ ) =            ml

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

Concentration of acid in Lead-acid battery  $N_2$  =

**Result:** the strength of acid in Lead –Acid battery is ----- M/lit

## EXPERIMENT-6

### Preparation of a polymer (Bakelite)

**Aim:** To prepare phenol-formaldehyde resin

**Apparatus:** Beakers, conical flask, glass rod, measuring cylinders, fractionation weight box etc.

**Chemicals:** glacial acetic acid, 40% formaldehyde solution, phenol, conc. HCl, distilled water.

**Theory:** phenol resins are condensation polymerization product of phenolic derivative with aldehyde (like formaldehyde). It is prepared by condensing phenol with formaldehyde in the presence of acid or alkaline catalyst.

#### Reaction:

Step 1: Formation of ortho and Para hydroxyl benzyl alcohols from phenol and formaldehyde.

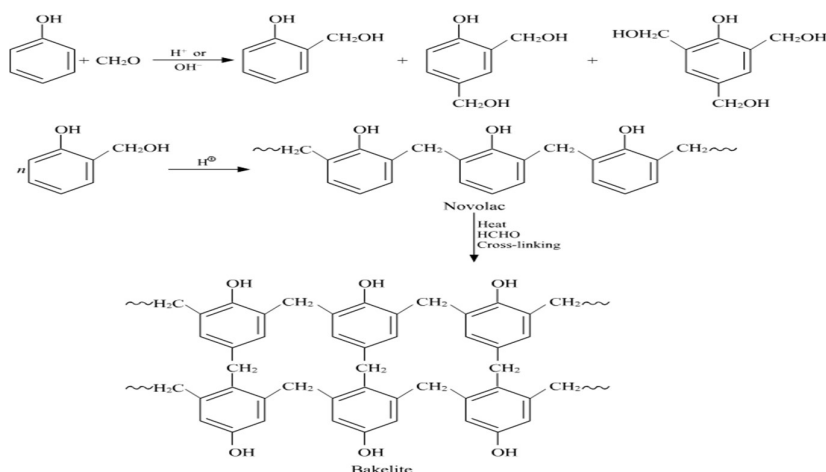
When phenol is treated with formaldehyde in the presence of acid it gives ortho and Para hydroxyl benzyl alcohols.

Step 2: formation of novolac:

When ortho hydroxyl benzyl alcohols are heated in the presence of acid it produces novolac.

Step 3: Formation of Bakelite

Further heating of novolac in the presence of hexamethylene tetramine produces hard, rigid, infusible polymer called Bakelite.





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### Procedure:

1. Place 5ml of glacial acetic acid and 2.5ml of formaldehyde solution in 500ml beaker.
2. Add 2gm of phenol and 1ml of conc.HCl solution in it.
3. Heat the solution slowly with constant stirring for 5mins.
4. A large mass of pink plastic is formed.
5. The residue obtained is washed several times with distilled waer.
6. Dry the product and calculate the yield accurately.

### Result:

The weight of obtained Bakelite is .....gms.





## EXPERIMENT-7

### Determination of percentage of Iron in Cement sample by colorimetry.

**Aim:** To estimate of  $\text{Fe}^{+3}$  iron present in the given sample of cement by colorimetry using ammonium thiocyanate as the reagent.

**Requirements:** HCl,  $\text{HNO}_3$  ammonium thiocyanate, cement Sample

**Principle:** The quantitative colorimetry is based on two principle laws of photometry. (i) Lambert's law, (ii) Beer's law. Ammonium thiocyanate produces blood red colour with ferric ion, and the colour produced is stable in Nitric acid. Its optical density is measured in a photocolourimeter and the concentration of ferric iron is obtained from a standard calibration curve.

#### Procedure

- (a) **To obtain standard calibration curve:** Dissolve the given ferrous ammonium sulphate in 100 ml of  $\text{H}_2\text{O}$  and add 5 ml of 1:5  $\text{H}_2\text{SO}_4$ , dil.  $\text{KMnO}_4$  solution through burette until light pink colour appears. Dilute the solution to 1 litre such that 1 ml of solution contains 0.1 mg of  $\text{Fe}^{+3}$ . From the above solution, take separately 1, 2, 3, 4, 5 ml into five 100 ml standard volumetric flasks. Add 1 ml of nitric acid and 5 ml of 40% ammonium thiocyanate solution to all the above samples to get blood red colour and made up the solutions to the mark by adding distilled water. Now measure the optical densities of all the solutions using photocolourimeter. Plot a graph by taking amount of ferrous iron on x-axis and optical density on the y-axis. The curve obtained is called standard calibration curve.
- (b) **Dissolution of sample:** Weigh about 0.1 gm of cement sample accurately and transfer into a clean and dry 250 ml beaker. Add about 5 ml of  $\text{H}_2\text{O}$  to moisten the sample. Place a glass rod and cover the beaker with a watch glass and add about 5 ml of concentrated HCl drop wise and heat the solution till the sample dissolves. Heat the beaker on small flame and evaporate the solution to almost dryness to expel the excess acid. Add about 20 ml of distilled water to the beaker to dissolve the contents. Then filter the solution through whatmann no. 47 filter paper into 100 ml standard volumetric flask.

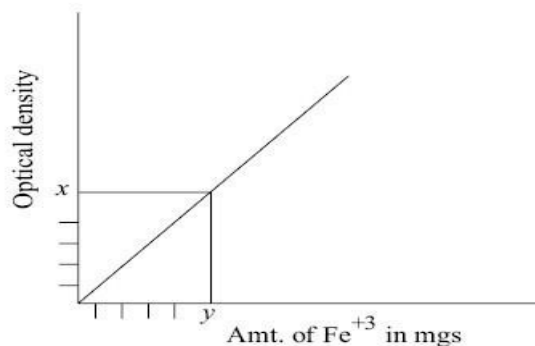
Wash the funnel with 10 ml portions of distilled water into the beaker, remove the funnel and make up the solution to 100 ml with distilled  $\text{H}_2\text{O}$ . Shake the flask well for uniform concentration.



### Development of Colour

Pipette out 10 ml of prepared solution into a 100 ml standard volumetric flask and add to it 1 ml conc.  $\text{HNO}_3$ . From the burette, add 5 ml of 40%  $\text{NH}_4\text{SCN}$  and make up the solution to 100 ml with distilled water and shake the flask well for uniform concentration. Measure the optical density of solution by using photocolorimeter and determine the concentration of iron from the calibration curve.

### Standard Calibration Curve



| conc. of $\text{Fe}^{+2}$ in mg. | Optical density |
|----------------------------------|-----------------|
| 0.05                             | 0               |
| 0.10                             |                 |
| 0.15                             | 0.32            |
| 0.20                             | 0.40            |
| 0.25                             | 0.50            |
| 0.30                             | 0.60            |
| 0.35                             | 0.70            |
| 0.40                             | 0.80            |

### Observation & Calculations

$$\text{Wt. of bottle + Cement sample} = W_1$$

$$\text{Wt. of empty bottle} = W_2$$

$$\text{Wt. of cement} = W_1 - W_2$$

$$\% \text{ Fe}^{3+} \text{ in the sample} = \frac{y \times 10 \text{ ml}}{(W_1 - W_2) \text{ mg}} \times 10$$

### Result

%  $\text{Fe}^{3+}$  is present in 0.1 gms of cement.....





## EXPERIMENT-8

### Estimation of calcium in port land cement

**Aim:** To determine percentage of CaO present in cement by using EDTA.

**Apparatus:** Burette, Burette stand, conical flask, pipette, Standard flask

**Chemicals:** EDTA, Patton and Reeder's Indicator, Cement sample, Hydrochloric acid, NaOH.

**Principle:** Cement contains silicates of iron, aluminum and calcium. Calcium is a prime constituent of cement. The general composition of a Portland cement is given here

| Constituent                          | Percentage |
|--------------------------------------|------------|
| CaO                                  | 66-66.6    |
| SiO <sub>2</sub>                     | 17.0-25    |
| Al <sub>2</sub> O <sub>3</sub>       | 03.0-8.0   |
| Fe <sub>2</sub> O <sub>3</sub>       | 2.0-6.0    |
| MgO                                  | 0.1-1.5    |
| Na <sub>2</sub> O & K <sub>2</sub> O | 1.0-3.0    |

In the estimation of calcium in cement, the given cement is treated with concentrated hydrochloric acid. The insoluble silica residue is removed by filtration. Iron and aluminium in the filtrate are converted as their hydroxides and filtered. Filtrate containing calcium ions is titrated at a pH of 12-14 against EDTA using Patton and Reeder's indicator. This indicator is used because Eriochrome Blank-T forms very weak complex with calcium ions. Di ethylamine is added to maintain a pH about 12 and glycerol is added to get a sharp end point. This method facilitates the determination of calcium only, although Mg<sup>2+</sup> ions are present in the cement solution which can be suppressed by adding NaOH.



### Procedure: Part A-Preparation of standard solution of disodium salt of EDTA.

Weigh accurately the given disodium salt of EDTA crystals using an electronic weighing balance. Note the weight, transfer the crystals carefully into a funnel placed over a 250 cm<sup>3</sup> volumetric flask and note down the empty weight of the weighing bottle. Add little water and 1/2 test tube of ammonia. Dissolve the crystals by swirling the flask gently. Add some more water if needed. Dilute the solution up to the mark with ion exchange water, stopper the flask and mix the solution thoroughly by inverting the flask several times so that a homogeneous solution results. Calculate the molarity of EDTA.

### Part B: Estimation of CaO

Pipette out 20 ml of the given cement solution into a clean conical flask. Add 2 cm<sup>3</sup> of 1:1 glycerol with constant shaking of the contents of the flask; add 2 cm<sup>3</sup> of diethyl amine and 1/2 test tube of 4N NaOH. Add 2-3 drops of Patton and Reeder's indicator. Titrate against the EDTA solution till the colour changes sharply from wine red to clear blue. Repeat the titration for concordant values.

| S.NO | VOLUME OF CALCIUM SOLUTION(V <sub>2</sub> ML) | BURETTE READINGS(ml) |       | VOLUME OF EDTA(V <sub>1</sub> ML) |
|------|---|----------------------|-------|-----------------------------------|
|      |   | Initial              | Final |                                   |
| 1.   |   |                      |       |                                   |
| 2.   |   |                      |       |                                   |
| 3.   |   |                      |       |                                   |

### Calculations:

Volume of EDTA solution (V<sub>1</sub>) =

Normality of EDTA solution (N<sub>1</sub>) =

Volume of calcium solution (V<sub>2</sub>) =

Normality of calcium solution (N<sub>2</sub>) =  $\frac{V_1 N_1}{V_2}$



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Weight of Cao in the given 250 ml of cement solution =

$$(a) \times \text{Eq. wt. of Cu (63.5)} \div 4 = \underline{\hspace{2cm}} \times 63.5 = \underline{\hspace{2cm}} \text{ g}$$



## EXPERIMENT-9

### Adsorption of acetic acid by charcoal.

**Aim:** To study the adsorption of acetic acid on charcoal and to verify Freundlich isotherm.

**Principle:** The phenomenon in which accumulation of other substances on the surface of a porous solid without a sensible penetration into the bulk of solid phase is called as adsorption.

At a given temperature, the variation in the amount of solute adsorbed with change in concentration of the solution is given by an empirical relation suggested by Freundlich known as Freundlich isotherm.

$$\frac{x}{m} = kc^{1/n}$$

where  $x$ -amount adsorbed by mg of adsorbent  
 $c$ -equilibrium concentration of adsorbate  
 $k$   $m$  are empirical constants.

### Procedure

Weigh accurately about 2 gms of finely powdered charcoal in each of the thoroughly cleaned and dried bottles numbered as 1 to 6. Prepare 0.5 M acetic acid solution for 250 ml by means of a burette. Add 10, 20, 30, 40, 50 ml of acid solution and 40, 30, 20, 10 and 0 ml distilled water in bottles 1, 2, 3, 4, 5 respectively

Take the given unknown solution in the bottle no. 6, shake the bottle vigorously and leave them at a desired temperature for about half an hour. Prepare 0.1 M NaOH for 250 ml and standardise with standard oxalic acid solution using phenolphthalein as indicator against standard NaOH then determine the concentration of acid solution.

Filter the solution of each bottle by means of filter paper. Collect the filtrates properly in flasks. Reject first few ml of filtrate in each case. Take 10 ml from each filtrate and then titrate with standard alkali. Calculate equilibrium concentration of the acid in each bottle.



Tabulate the observations and result. Plot a graph between  $\log x/m+2$  values and  $\log C_2 + 2$  values.

### Observation & calculators

| S .<br>No. | $C_1$ | $C_2$ | $\log C_1$ | $\log C_2$ | $x = C_1 - C_2 \times \frac{126}{10}$ | $\frac{x}{m}$ | $\log \frac{x}{m}$ | $\log \frac{x}{m} + 2$ | $\log C_2 + 2$ |
|------------|-------|-------|------------|------------|---------------------------------------|---------------|--------------------|------------------------|----------------|
|            |       |       |            |            |                                       |               |                    |                        |                |

**Result:** Hence the Freundlich adsorption isotherm is verified.



## EXPERIMENT-10

### Determination of percentage Moisture content in a coal sample

**Aim:** To determine the moisture content in a given coal sample

**Theory:** Coal is fossil fuel which occurs in large the earth crust. It was been formed by the partial decay of plant materials accumulated millions years and further altered by action of heat and pressure. Coal is highly carbon matter and important fuel. The process of conversion of wood into coal be represented as

Wood ----- Peat -----Lignite----- Bituminous----- Anthracite

The coal is classified according to rank which measure of change. It has underground in the transition from wood to anthracite rank based on carbon content. The composition of coal varies widely and hence it is necessary to analyze and interpret the result from the point of commercial classification , the price fixation and proper industrial utilize, the following methods are analyze can be used for moisture , content in coal. .

#### PROXIMATE ANALYSIS

This includes the determination of moisture volatile matter and ash and fixed carbon content, which give value information regarding the commercial classification. This is known as proximate analysis.

#### ULTIMATE ANALYSIS:

Which includes the estimation of ash, carbon, hydrogen, sulphur, nitrogen and oxygen? The ultimate analysis is essential for calculating heat balance in any process for which coal is employed as fuel.

| Sample (W3) | Mass of crucible | Weight of Crucible along with substance<br>(Before Heating) W1 | Weight of crucible along<br>with substance (After<br>heating) W2 |
|-------------|------------------|--|--|
| 1           |                  |  |  |
|             |                  |  |  |
|             |                  |  |  |
|             |                  |  |  |





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### Calculation of moisture content:

Calculate the present of moisture in the analysis in the sample as follows

% Moisture content =  $\frac{W1-W2}{W3} \times 100$  where

W1= mass of the crucible and sampler

W2=mass of crucible

W3= mass of sample in grams

Significance of proximate analysis:

Moisture

Increase the transport cost

Reduce the caloric value

Considerable amount of heat is wasted in evaporating the moisture during the combustion of moisture.  
Hence, high percentage of moisture is undesirable.

### Result :

Moisture content = 40 %





## EXPRIMENT-11

### DETERMINATION OF VISCOSITY OF OILS USING REDWOOD VISCOMETER I AND II

**Aim:** Determination of viscosity of lubricant by Redwood viscometer (No. 1 & 2).

**Apparatus required:** Redwood viscometer (No. 1 & 2), stop watch, thermometer, given lubricant oil and distilled water.

#### *Theory*

Viscosity is defined as the internal friction offered by internal friction offered by the layers of fluid of its flow. Viscosity is a measure of flow ability of a liquid at a definite temperature. It determines the performance of oil under operating conditions. Higher the viscosity of fluid lesser will be its flow.

Coefficient of viscosity is called absolute viscosity is defined as tangential force per unit area required to maintain a unit velocity gradient between two parallel layers a unit apart. It is denoted by  $\eta$  (eta).

Mathematically,  $\eta =$

Where  $F =$  force  $A =$  Area

$dv/dx =$  velocity gradient.

**Units:** In C.G.S. system poise =  $\text{dyne cm}^{-1}$

$^2\text{s}$  In SI system  $\text{Nm}^{-2}\text{s}$ .

The absolute viscosity of lubricant is determined by measuring the time of flow of the oil through a capillary of definite dimensions at uniform temperature. The viscosity is can be measured by a Redwood viscometer.

#### ***Description of redwood viscometer:***

It is available in two sizes. These are:

- (i) RW<sub>1</sub> or Redwood No. 1 (Universal)
- (ii) RW<sub>2</sub> or Redwood No. 2 (Admiralty)

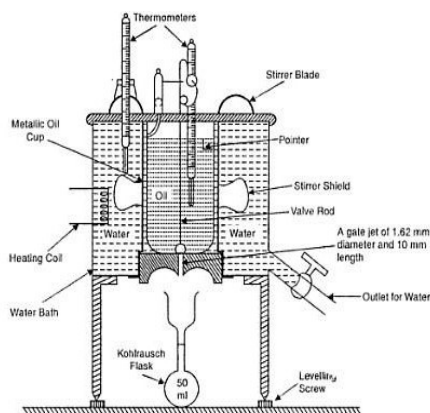
| Viscometer      | Diameter of capillary | Length of jet |
|-----------------|-----------------------|---------------|
| RW <sub>1</sub> | 1.62 mm               | 10 mm         |
| RW <sub>2</sub> | 3.8 mm                | 50 mm         |

The rate of discharge of oil through RW<sub>2</sub> is nearly 10 times faster than the discharge through RW<sub>1</sub> because receiving flask of RW<sub>2</sub> has a wider mouth.

### Construction

It consists of a standard brass oil cup fitted with an agate jet of specific dimension in the middle of base and open at the upper end (Fig. 1). The jet can be opened or closed by a polished ball valve which controls the flow of oil. A pointer is provided in the oil cup to indicate the level up to which oil shall be filled in it. The lid of the cup is provided with a thermometer to note the temperature of oil. Surrounding the oil cup is cylindrical vessel made of copper. This vessel is filled with water and serves as a water bath to maintain the oil at a desired temperature of oil. It is heated by means of heating coils.

The water bath is provided with stirrer having four blades to maintain uniform temperature of bath. A thermometer is fitted in a water bath to know the temperature of water at its base. A 50 mL flask called Kohlrusch flask is provided below the jet to collect the liquid flowing out of the jet. The apparatus is also provided with leveling screws for its leveling.



**Fig. 1.** Redwood apparatus no. 1 & 2.



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### Procedure:-

1. Level the viscometer with the help of leveling screws. Fill the outer bath with water and connect to the electric mains. Clean the oil cup and discharge jet with a suitable jet with a suitable solvent like hexane, carbon tetrachloride etc. and properly dry it.
2. Place the ball valve on agate jet to close it. Pour the test oil in the cup carefully up to the pointer. Insert a thermometer and stirrer and cover the lid.
3. Adjust the temp. of water bath until the oil attains the desired temperature. In this period keep water in water bath and oil in oil cup.
4. Place a clean and dry Kohlrausch flask immediately below and directly in the line with the discharge jet
5. Remove the ball when oil attains desired temperature with one hand and start stop watch with the other hand. Allow the oil to flow till the flask is filled up to 50 mL mark. Stop the stop watch and note the time of flow in seconds.
6. Repeat the experiment 3-4 times and record the reading.
7. Report the mean value in Redwood seconds and also mentioning the viscometer used and the test temperature.

### Observations

**Table: For time of flow**

| Sr. No. | Temperature (°C) | Time of flow (RW seconds) |
|---------|------------------|---------------------------|
| 1       |                  |                           |
| 2       |                  |                           |
| 3       |                  |                           |

**Result:** Viscosity of given lubricant is .....RW seconds.



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## EXPRIMENT-12

### DETERMINATION OF CALORIFIC VALUE OF GASEOUS FUELS BY JUNKER'S CALORIMETER

**Aim:** to determine the calorific value of gaseous fuel by Junker's gas calorimeter.

**Apparatus:** Junker's gas calorimeter assembly, gas flow meter, a source of continuous supply of water, four thermometers, a gas source whose calorific value is to be determined.

#### Theory:

Calorific value, also called heating value, is defined as the quantity of heat liberated by the complete combustion of unit quantity of fuel. There are two types of calorific values, higher or gross calorific value and the lower or net calorific value.

1. Higher or gross calorific value may be defined as the total amount of heat liberated when one unit of the fuel is burnt completely and the combustion products are cooled to room temperature.
2. Lower or net calorific value, may be defined as the amount of heat liberated when one unit of the fuel is burnt completely and the combustion products are allowed to escape. higher or gross calorific value and lower or net calorific value are thus related as:

**Net calorific value=gross calorific value-latent heat of water vapor formed.**

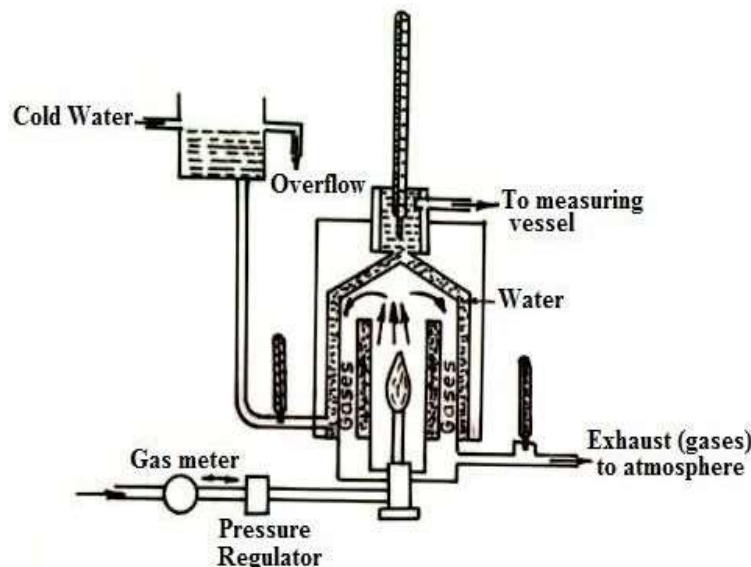
Since one part by weight of hydrogen gives nine parts by weight of water the above equation becomes,

**Net calorific value=gross calorific value-(weight of H<sub>2</sub> x 9 x latent heat of steam)**

The apparatus used for determining the calorific value is called calorimeter. It operates on the principle that heat liberated from a known quantity of fuel, burnt in an atmosphere of pure oxygen in the sealed and thoroughly insulated chamber of calorimeter, can be completely utilized for raising the temperature of the calorimeter and a known quantity of water in the calorimeter. By considering that the heat absorbed by the calorimeter and water and the heat given out by the fuel are equal the calorific value of the fuel can be determined.

## Description:

Junker's gas calorimeter consists of an inner combustion chamber in which the gas burns. This chamber is surrounded by a water jacket through which a number of tubes, called flues, pass. Water enters the calorimeter at a constant heat and steady flow rate. Its inlet and outlet temperatures are recorded. The gas whose calorific value to be determined is made to pass through a gas meter, which records its volume at room temperature and burns in the inner combustion chamber. The gas on combustion heats the water jacket and then passes down the flues in the water jacket, thus complete transfer of the heat of combustion to water is ensured. The hot products of combustion move upward in the chamber and then downward through the flues provided in the water jacket and escape through the exit. A thermometer at the exit indicates its temperature, which should be as near the room temperature as possible so that gases in the flues, goes out from the upper portion. Its temperature and volume are recorded. Water formed by the condensation of steam is also collected. Knowing the weight of the water flowing and the rise in its temperature, the heat given out by the gas is known.





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Approved by AICTE NEW DELHI & Affiliated to JNTU-GV, Vizianagaram  
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### Procedure:

The Junker's gas calorimeter is placed on a flat rigid platform near an uninterrupted continuous water source. The gas source is connected to the pressure regulator, gas flow meter and the burner respectively in series. The thermometers are inserted in to their respective places to measure the temperature of flue gas at the flue gas outlet. The water flow is started through the calorimeter at a steady flow rate and allowed it to drain through over flow.

The gas flow is started slowly and the burner is ignited outside the calorimeter. Gas flow is regulated at a steady rate. Burner is inserted now in to the calorimeter and allowed the outlet water temperature to attain steady state.

2000ml measuring jar is kept beneath the swinging water outlet tube and the stop watch is simultaneously started and the initial gas flow meter reading is noted. The time taken to fill 1000ml is noted and at the same time the final gas flow reading is recorded by the gas flow meter.

All the readings are tabulated and the calorific values of the gas under test is calculated.





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Table

| s.no | Volume of water collected in lit.<br>( $V_w$ ) | Volume of gas burnt in lit.<br>( $V_g$ ) | Water inlet temperature<br>$T_1$ °C | Water outlet temperature<br>$T_2$ °C | Change in temperature of water<br>$\Delta T = (T_2 - T_1)$ | CV of gas in Kcal/kg |
|------|--|--|-------------------------------------|--------------------------------------|--|----------------------|
|      |  |  |                                     |                                      |  |                      |
|      |  |  |                                     |                                      |  |                      |





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### Observations:

Density of water,  $\rho_w = 1000 \text{ kg/m}^3$

Volume of gas burnt,  $V_g$  in lits =

Density of gas,  $\rho_g = 0.22 \text{ kg/m}^3$

$C_{p_w} = 1 \text{ k cal/kg K}$

Time taken to collect 1lit of water = .....sec.



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## Calculation

$$CV_{\text{gas}} = \frac{V_w \times \rho_w \times C_{p_w} \times \Delta T}{V_g \times \rho_g}$$

Where

$\rho_w$  = density of water

$V_g$  = volume of gas burnt in lit

$\rho_g$  = density of gas

$C_{p_w}$  = specific heat of water

**Result:**

Calorific value of the given gaseous fuel is..... k cal/kg.



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