SVR ENGINEERING COLLEGE

(Ayyaluru Metta(V), Nandyal)
(Affiliated to JNTUA, Ananthapuramu & Approved by AICTE, New Delhi)
ANDHRA PRADESH-518502

2021-22

ENGINEERING CHEMISTRY LABORATARY MANUAL

(20A51201P) – ME & CE

Prepared by

Dr. S SREENIVASULU Associate Professor
Mr M SHANOOR BASHA Assistant Professor
Mr. M SREENIVASULU Assistant Professor
Mr P PARABRAHAMAM Assistant Professor
Ms P HIMA BINDU Assistant Professor

For

I B.TECH

DEPARTMENT OF HUMANITIES & SCIENCE

CERTIFICATE

This is to certify that Mr./Miss……………………….………...

Registered No……………………………..……..………… has successfully completed the Experiments provided in the Applied Physics Laboratory developed by the Department of Physics, SVR Engineering College, Nandyal prescribed for the First Year B.Tech Courses, and as Approved by the Jawaharlal Nehru Technological University, Anantapur for the year …………………….

(Signature)
Head of the Department

(Signature)
Faculty In-charge

STUDENT PROFILE

Name of the Student : --

Regd. Number : --

Branch / Section : ---

Contact Phone No : --

E-mail ID : ---

Father's Name : ---

Father's Contact No : ---

Residential Address : --

VISION OF THE COLLEGE

To produce competent engineering graduates & Managers with a strong base of Technical & Managerial knowledge and the complementary skills needed to be successful professional engineers & Managers.

MISSION OF THE COLLEGE

To Fulfill The Vision By Imparting Quality Technical & Management Education To The Aspiring Students, By Creating Effective Teaching / Learning Environment And Providing State-Of The –Art Infrastructure And Resources.

DEPARTMENT OF HUMANITIES AND SCIENCES

VISION

To build foundation for excellence and spur development of the Institution as a premier Institution by igniting and nurturing enthusiasm, interests and passion in the study of Mathematics, Physics, Chemistry & English in professional courses, as a part of curriculum.

MISSION

- To awaken the young minds and discover their talents both in theory and in practical Physics through dedicated teaching, commitment to students and innovative instructional methods.
- To support the developmental activities of the College and make the department vibrant.
- To make vital contributions in areas of emphasis such as faculty, modern labs, Department library and demonstrate a high level of competence in the study of Mathematics, Physics, Chemistry & English
- To introduce pioneering programs in the Department that will embrace heritage and values of the Institution.
- To organize and sustain efficient operating systems in the Department for realization of our objectives as Institution of eminence and International standards.
- To evolve strategies in the Department for continuous Improvement.

PROGRAMME OUTCOMES

By the end of completing 4 years full time B.Tech in CSE, Graduates of B.Tech will be able to:

PO 1.Engineering Knowledge: Apply the knowledge of mathematics, science, engineering fundamentals, and an engineering specialization to the solution of complex engineering problems.

PO 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.

PO 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.

PO 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.

PO 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.

PO 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.

PO 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.

PO 8.Ethics: Apply ethical principles and commit to professional ethics and responsibilities and norms of the engineering practice.

PO 9. Individual and Team Work: Function effectively as an individual, and as a member or leader in diverse teams, and in multidisciplinary settings.

PO 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.

PO 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.

PO 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.

Chemistry laboratory outcomes (20A51201P)

S. No	LAB outcomes		Cognitive Process Dimension
1	**CO1**	Determine the cell constant and conductance of solutions	**Apply**
2	**CO2**	Prepare advanced polymer materials	**Understanding**
3	**CO3**	Determine the physical properties like surface tension, adsorption and viscosity	**Apply**
4	**CO4**	Estimate the Iron and Calcium in cement	**Apply**
5	**CO5**	Calculate the hardness of water	**Analyzing**

JAWAHARLAL NEHRU TECHNOLOGICAL UNIVERSITY ANANTAPUR

B.Tech – I/II Sem **L T P C**

0 0 3 1.5

(20A51201P) Engineering Chemistry Lab

(CIVIL & MECH)

List of Experiments:

1. Determination of Hardness of a groundwater sample.
2. pH metric titration of (i) strong acid vs. strong base, (ii) weak acid vs. strong base
3. Determination of cell constant and conductance of solutions
4. Potentiometry - determination of redox potentials and emfs
5. Determination of Strength of an acid in Pb-Acid battery
6. Preparation of a polymer
7. Determination of percentage of Iron in Cement sample by colorimetry.
8. Estimation of calcium in port land cement.
9. Adsorption of acetic acid by charcoal.
10. Determination of percentage Moisture content in a coal sample.
11. Determination of Viscosity of lubricating oil by Red Viscometer 1 &2.
12. Determination of calorific value of gaseous fuels by JUNKE’S calorimeter.

Course Outcomes:

At the end of the course, the students will be able to

- **determine** the cell constant and conductance of solutions (L3)
- **prepare** advanced polymer materials (L2)
- **determine** the physical properties like surface tension, adsorption and viscosity (L3)
- **estimate** the Iron and Calcium in cement (L3)
- **calculate** the hardness of water (L4)

ADDITIONAL EXPERIMENTS

S. No	Name of the experiment
1	Introduction of chemistry laboratory and safety guide lines
2	Estimation copper by idometric method
3	Estimation of Zinc by EDTA method

Introduction of engineering chemistry laboratory:

The Engineering Chemistry Lab has a wide range of equipment. The lab is aptly prepared to impart education in Chemistry in a neatly designed, spacious and well-ventilated laboratory with a capacity to accommodate 30 students. The lab is aesthetically designed with polished ceramics tiles.

It provides students with a practical approach towards the various techniques used in engineering application. Practical awareness is inculcated and students are trained both quantitatively and qualitatively during the lab sessions so that their understanding and problem solving abilities can be enhanced.

Applications of chemistry laboratory:

1. It is used to measure the strength of acids in secondary cells, ex: Lead-acid battery
2. It is used to measure the equivalent or end point for all types of volumetric titrations
3. It is used to measure the conductance and potential for all types of volumetric titrations.
4. It is used polymer materials and it’s engineering applications.
5. It is used to determine hardness of water.
6. It is used to determine moisture content in coal.

SCHEME OF VALUATION

Internal Lab Exam

1.Day to day Observation : 10 M

2. Day to day performance : 10M

3.Day to day Record : 10 M

Final Internal Lab Exam : 30 M

Total Internal Marks : 30 M

External Lab Exam

1. Record : 10 M
2. Write up : 25 M
3. Experiment : 15 M
4. Result : 10 M
5. Viva-Voce : 10 M

Total External Marks : 70 M

Total Final Lab Marks : 70 M

WRITE UP PROCEDURE

1. AIM AND FORMULA WITH TERMS EXPLANATION
2. PROCEDURE
3. TABULAR FORMS
4. MODEL GRAPH
5. PRECAUTIONS
6. CALCULATIONS
7. RESULT

Rules to be followed in Chemistry Laboratory

1. Student should wear Apron whenever they enter the laboratory for practice.
2. Student should wear Identification card (ID).
3. Student should come to the lab along with Observation book and Record.
4. While entering and leaving the lab, student should sign in the log book.

Experment-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 Ca^{2+} and Mg^{2+} ions which forms wine red color complex with the indicator.

Ca^{2+} (or) Mg^{2+} + EBT ———> Ca-EBT (or) Mg-EBT

(Wine red color complex)

EDTA forms a color less complex with the metal ions (Ca^{2+} andMg^{2+})

Ca-EBT (or) Mg-EBT + EDTA———> Ca-EDTA (or) Mg-EDTA + EBT

(Wine red color 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:

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

Normality of EDTA (N1) = $\frac{\text{Weight of substance X100}}{\text{Equivalent EDTA X volume of solution (100ml)}}$ = 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_1) = ml
Molarity of EDTA solution (M_1) =
Volume of water solution (V_2) = 20 ml
molarity of water solution (M_2) = =

Hardness of given ground water sample= N2X100X1000 ppm

RSULT

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.

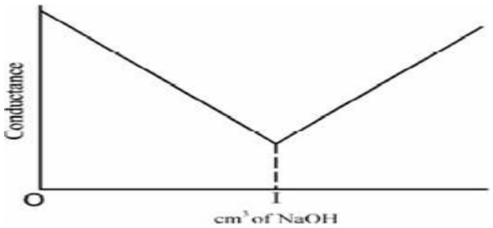
CHEMICALS REQUIRED: Sodium hydroxide, Hydrochloric acid

PRINCIPLE: At first solution contain H^+ and Cl^- ions. Since H^+ ions 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^+ ions do not process much mobility. As the neutralization point and solutions 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.



Tabular form and calculations:

S. No	Volume of NaOH	Observed conductance
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		
21		
22		
23		
24		
25		

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,

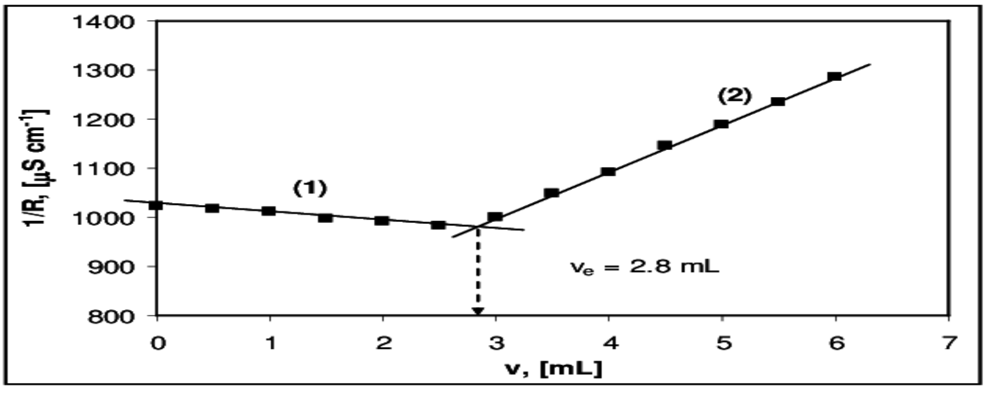
PRINCIPLE: At first solution contain $CH_3COO^-$and H^+ ions. Since H^+ ions posses greater mobility it follows that the conductivity is mainly due to H^+ ions. The addition of NaOH is represented by the equation.

$$H^+ + CH3COO^- + Na^+ + OH^- \longrightarrow CH_3COONa + H_2O$$

As NaOH is added the H^+ ions are removed. The conductivity decreases as Na^+ ions do not process much mobility. As the neutralization point and solutions 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 CH_3COOH is prepared. 20 ml CH_3COOH of 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.

Graph :



Tabular form and calculations:

S. No	Volume of NaOH	Observed conductance
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		
21		
22		
23		
24		
25		

FORMULA: $N_1V_1 = N_2V_2$

RESULT: The normality of weak (**CH_3COOH**) 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 cleansingacid, 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 touchthe 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 fifteenminutes 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 x 1000/C

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 (λ)	

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 from,
- 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 potentometric 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 tirant. Here the end pint 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 from. 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.

$Cr_2O^{2-}{}_7 + 14H + 6e \rightleftharpoons 2\ Cr^{3+} + 7\ H_2O$ ... (1)

While Fe2+ which is used to titrate $K_2Cr_2O_7$ gets oxidised to Fe3+ as per the reaction

Fe2+ ⇌ Fe3+ + e … (2)

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

$Cr_2O_{2-7} + 6\ Fe_{2+} + 14H_{+} \rightleftharpoons 6\ Fe_{3+} + 2Cr_{3+} + 7\ H_2O$ ... (3)

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

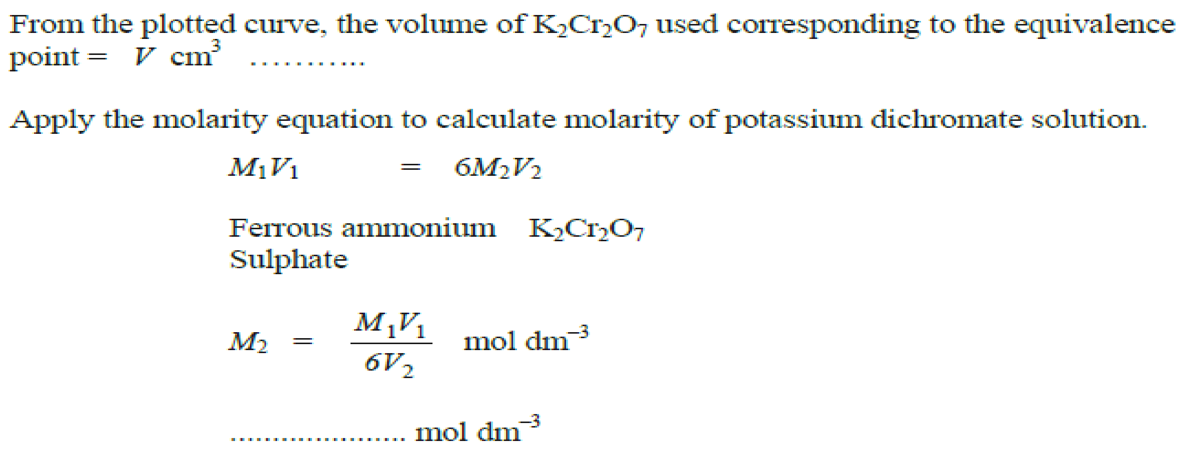
PROCEDURE

1. Using 100 cm3 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 cm3 of given Fe2+ solution and add 25 cm3 dilute H2SO4 acid and 50 cm3 of distilled water in a 250 cm3 beaker.
3. SCE is used as the Reference electrode. Platinum metal foil, dipped in Fe2+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 cm3 of 0.02 M K2Cr2O7 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 cm3 of K2Cr2O7 at a time and go on noting the E.M.F. after each addition.
8. When the volume reached near about 1 cm3 of the expected equivalence point (approximate), add the solution from burette in 0.5 cm3 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.

Observations:-

S. No	**Volume (*V*) of K2Cr2O7 added (cm3)**	**EMF. of (*E*) the cell (millivolts)**	**Δ *V***	**Δ *E***	**Δ *E*/ Δ *V***

CALCULATIONS:-



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

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

$$M_1V_1 \quad = \quad 6M_2V_2$$

Ferrous ammonium Sulphate $K_2Cr_2O_7$

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

.... 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.

$$H_2SO_4 \quad + \quad NaOH \quad \rightarrow \quad Na_2SO_4 \quad + \quad H_2O$$

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

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

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.

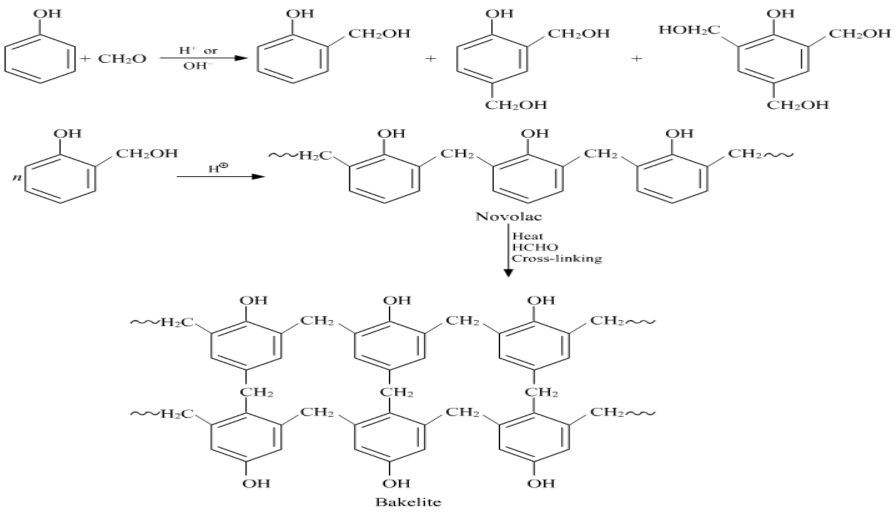
When phenol is treated with formaldehyde in the presence of acid it gives ortho and Para hydroxyl benzyl alcohols.

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.



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 Fe^{+3} iron present in the given sample of cement by colorimetry using ammonium thiocyanate as the reagent.

Requirements:HCl , 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 photocolorimeter 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 H_2O and add 5 ml of 1:5 H_2SO_4, dil. $KMnO_4$ 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 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 photocolorimeter.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.

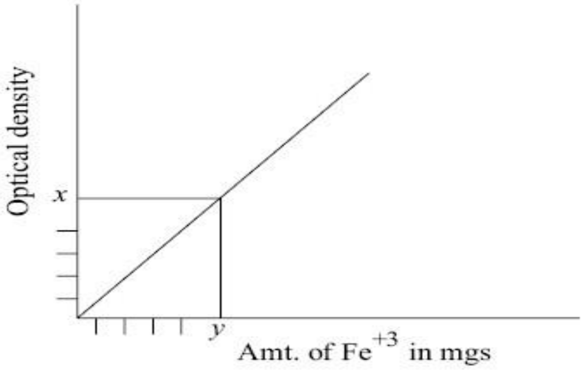
(b) **Dissolution of sample:** Weigh about 0.1 gm of cement sample accurately and tra fer into a clean and dry 250 ml beaker. Add about 5 ml ofwater$_2$O to moisten the sample. Place a glass rod and cover the beaker with a watch glass and add about 5 ml of concentrated HCI drop wise and heat the solution 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 H_2O. 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. HNO_3. From the burette, add 5 ml of 40% NH_4SCN 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 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

% 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_2	17.0-25
Al_2O_3	03.0-8.0
Fe_2O_32	2.0-6.0
MgO	0.1-1.5
Na_2O & K_2O	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 $Mg2+$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 cm3volumetric 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 cm3 of 1:1 glycerol with constant shaking of the contents of the flask; add 2 cm3of diethyl amine and ½ test tube of 4N NaOH. Add 2-3drops 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(V2ML)	BURETTE READINGS(ml)		VOLUME OF EDTA(V1ML)
		Initial	Final	
1. 2. 3.				

Calculations:

Volume of EDTA solution (V_1) =

Normality of EDTA solution (N_1) =

Volume of calcium solution (V_2) =

Normality of calcium solution (N2) = $\frac{V1N1}{V2}$

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

(a)× Eq. wt. of Cu (63.5) 4= _____×63.5= ________ 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 emperical relation suggested by Freundlich known as Freundlich isotherm.

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

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 powderd 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 receptively

Take the given unknown solution in the bottle no. 6, shake the bottle vigorously and leave them at a desired temperature for about half as hour. Prepare 0.1 M NaOH for 250 ml and standardise with standard oxalic acid solution using phenolpthaline 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 filterate and then titrate with standard alkali. Calculate equilibrium contcentration 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. 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 fussily 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 (Before Heating) W1	Weight of crucible along with substance (After heating) W2
1			

Calculation of moisture content:

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

% Moisture content =W1-W2/W3X100 where

W1= mass of the crucible and sampler

W2=mass of crucible

W3= mass of sample in grams

Singinfincane of proximate analysis:

Moisture

Increase the transport cost

Reduce the caloric value

Considerable amount of heat is wasted in evaporating the moisture during the combusted of moision . 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 η (eta).

Mathematically, η =

Where F = force A= Area

dv/dx=velocity gradient.

Units: In C.G.S. system poise = dyne
$cm^{-2}s$ In SI system $Nm^{-2}s$.

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_1 or Redwood No. 1 (Universal)

(ii) RW_2 or Redwood No. 2 (Admiralty)

Viscometer	Diameter of capillary	Length of jet
RW_1	1.62 mm	10 mm
RW_2	3.8 mm	50 mm

The rate of discharge of oil through RW_2 is nearly 10 times faster than the discharge through RW_1 because receiving flask of RW_2 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 Kohlrausch 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.

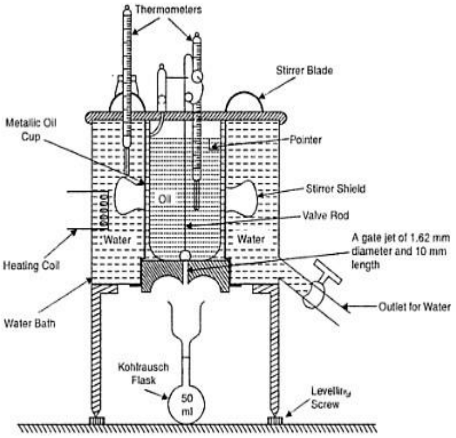


Fig. 1. Redwood apparatus no. 1 & 2.

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 (^oC)	Time of flow (RW seconds)
1		
2		
3		

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

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.

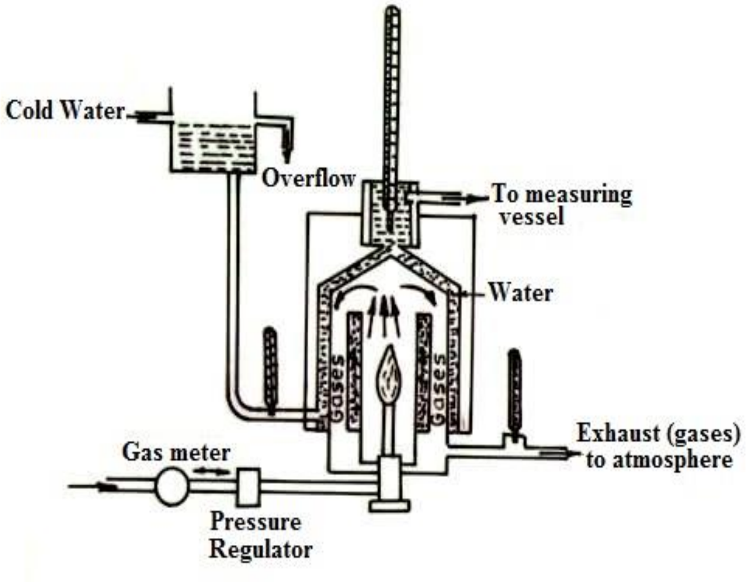
Since one part by weight of hydrogen gives nine parts by weight of water the above equation becomes,

Net calorific value=gross calorific value-(weight of H_2 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 completer 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.



Procedure:

The Junker’s gas calorimeter is placed on a flat rigid platform near an uninterrupted continues 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 startred through the calorimeter at a steady 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.

Table

s.no	Volume of water collected in lit. (V_w)	Volume of gas burnt in lit. (V_g)	Water inlet temperature $T_1{}^0C$	Water outlet temperature $T_2{}^0C$	Change in temperature of water $\Delta T=(T_2-T_1)$	CV of gas in Kcal/kg

Observations:

Density of water, ρ_w=1000 kg/m^3

Volume of gas burnt, V_g in lits =

Density of gas,ρ_g=0.22kg/m^3

Cp_w= 1k cal/kg K

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

Calculation

$$CV_{gas} = \frac{V_w \times \rho_w \times Cp_w \times \Delta T}{V_g \times \rho_g}$$

Where

ρ_w = density of water

V_g = volume of gas burnt in lit

ρ_g = density of gas

Cp_w = specific heat of water

Result:

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