TULSIRAMJI GAIKWAD-PATIL College of Engineering and Technology
Wardha Road, Nagpur - 441108
Accredited with NAAC A+ Grade
Approved by AICTE, New Delhi, Govt. of Maharashtra
(An Autonomous Institute Affiliated to RTM Nagpur University)
Department of Basic Sciences & Humanities

Date_____

Experiment No.1

Aim: **To determine percentage of Moisture ,Volatile matter and Ash content in a given coal sample. [CO1]**

Apparatus: Silica crucible, coal sample, Desiccators, muffle furnace, oven, digital balance and weight box.

Theory:

Coal is a fossil fuel, which occurs in levels in the earth crust, it has been formed by the partial decay of plant material accumulated millions of years ago and further attended by the action of heat and pressure. The analysis of coal is important for its commercial classification, price fixation and proper industrial utilization.   The quantity of a coal is ascertained by the following two types of analysis.

1) **Proximate analysis:** It includes the determination of moisture, volatile matter, ash and fixed carbon .This gives quick and valuable information regarding the commercial classification and determination of suitability for particular industrial use.

2) **Ultimate analysis:** It includes the estimation of carbon hydrogen, sulphur, nitrogen, oxygen and ash. The ultimate analysis is useful in combustion calculation (i.e. for calculation heat balance in any process for which coal is used as fuel)

Procedure for Proximate analysis:

Determination of Moisture content:

Weigh the silica crucible + add 1 gm of the air dried coal sample in it and again weight it accurately. Heat the crucible in an oven at a temperature of 105 -110° C for one hour. Cool the crucible first in air then in desiccators and weigh it again. The process of heating, cooling and weighing is repeated till a constant weight is obtained. The loss in weight of coal gives total moisture content. By knowing the loss in weight of coal, the percentage of moisture can be calculated as follows.

Determination of volatile matter:

Then put the same crucible in muffle furnace with lid for 7 minutes at 900-925° C take out the crucible and cool it first in air and then in a desiccators and again take its weight. By knowing the weight of volatile matter removed its percentages can be calculated by subtracting the percentage of moisture, which gives the volatile matter.

Determination of Ash content:

Then put the same crucible for one hour at 750° C without lid in muffle furnace so as to complete the combustion. Remove the crucible from the furnace, cool it in air and then in desiccators and weigh it. The weight of residue left in the crucible corresponds to the ash content of the coal.

Significance:

The composition of coal varies widely and hence it is necessary to analyze and interpreted the result from the point of view of commercial classification, price fixation and proper industrial utilization. Each constituent determined under the proximate analysis has its own implication and importance in the assessment of coal sample.

Volatile matter:- The volatile matter is not a constituent of coal but consist of   complex mixture of gaseous and liquid products resulting from the thermal decomposition of the coal substance. The volatile matter content of the coal is related to the length of flame, smoke forming tendency and the ignition characteristic High smoke and relatively low heating value. Coal with low volatile content burns with shorter flame .Thus higher the volatile matter content the larger is the combustion space required. Hence the volatile content of the coal influences the furnace design.

Ash:- The nature of the ash and its amount in a cool and softening temperature are very important in determining the quality of coal. Ash reduces the heating value of a cool. Ash usually consists of silica, Alumina, iron oxide and small quantities of lime and magnesia etc. its composition of considerable importance in metallurgical operation as it affects the slag and metal

composition and consequently is a prime consideration in selecting flux. The nature of ash and its amount in coal are very helpful in deciding the quality of coal. Hence, lesser the ash content better is the quality of coal as fuel

Fixed carbon:- The fixed carbon content increases from low ranking coal such as lignite to high –ranking coal such has anthracite. It the fixed carbon which burns in the solid state. Hence information regarding the percentage of fixed carbon in helps in designing the furnace and the firebox.

Observation and calculation:

Determination of moisture content

Weight the silica crucible= W_1 =……………..g

Weight of crucible + coal sample= W_2 =……….g

Weight of coal sample taken= (W_2-W_1)=…………g

Weight of crucible + coal after heating and cooling = W_3 =……..g

loss in weight of coal =(W_2-W_3) =………….g

$$\% \ of \ moisture \ content = \frac{W_2 - W_3}{W_2 - w_1} \times 100$$

Weight of crucible + coal after heating at 925°C for 7 min = W_3 =………...g

Loss in weight of or weight volatile matter removed = (W_2-W_3) =…………. g

$$\% \ of \ volatile \ matter \ = \left[\frac{W_2 - W_3}{W_2 - w_1} \times 100\right] - \% \ moisture \ content$$

Determination of Ash content-

Weight the silica crucible= W_1=……………..g

Weight of crucible + coal sample= W_2 =……….g

Weight of coal sample taken= (W_2-W_1) =…………g

Weight of crucible + coal after heating at 750°C for 1 hour = W_3 =………...g

Weight of ash formed = (W_3-W_1) =…………g

$$\% \ of \ Ash = \frac{Wt \ of \ Ash \ formed}{Wt \ of \ powdered \ coal \ taken} \times 100$$

$$\% \ of \ Ash = \left[\frac{W_3 - W_1}{W_2 - W_1} \times 100\right]$$

Determination of fixed carbon %:

% of fixed carbon residue = 100 - % (moisture + volatile matter + ash)

Result:

In the given coal sample, % of moisture =…………………. %

In the given coal sample, % of volatile matter=……………%

% of Ash =……………%

% of fixed carbon=………………%

Course Outcome:

	Course Outcome	PO
CO 1	**Interpret** the types of Energy sources and its properties and application.	PO1, PO2, PO6, PO10, PO12

PO Mapping:

Course Outcome	Program Outcomes											
	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12
CO 1	2	1	-	-	-	1	-	-	-	1	-	2

Viva Questions:

1. What is fuel? How many types of fuel are known to you?
2. What is coal? How is coal formed?
3. Why is it necessary to analyze coal sample?

4. What are the types of coal analysis?

5. Why should be the moisture content of coal be low?

6. Which apparatus is used for moisture analysis? At what temperature is moisture analysis done and why?

7. What is calorific value of fuel?

8. What are the requirements of good quality fuel?

9. What is moisture?

10. Why the analysis of coal should be done at 105^0C to 110^0C?

11. What are the different types of coal?

12. What is volatile matter in coal?

13. What is the temperature for determination of volatile matter?

14. Which apparatus is used to determine volatile matter?

15. What is the use of high volatile matter containing coal? What is Coke

Conclusion: Thus, we have studied the determination of Moisture content ,volatile matter and ash in a coal successfully.

Signature of Lab Course Coordinator_____

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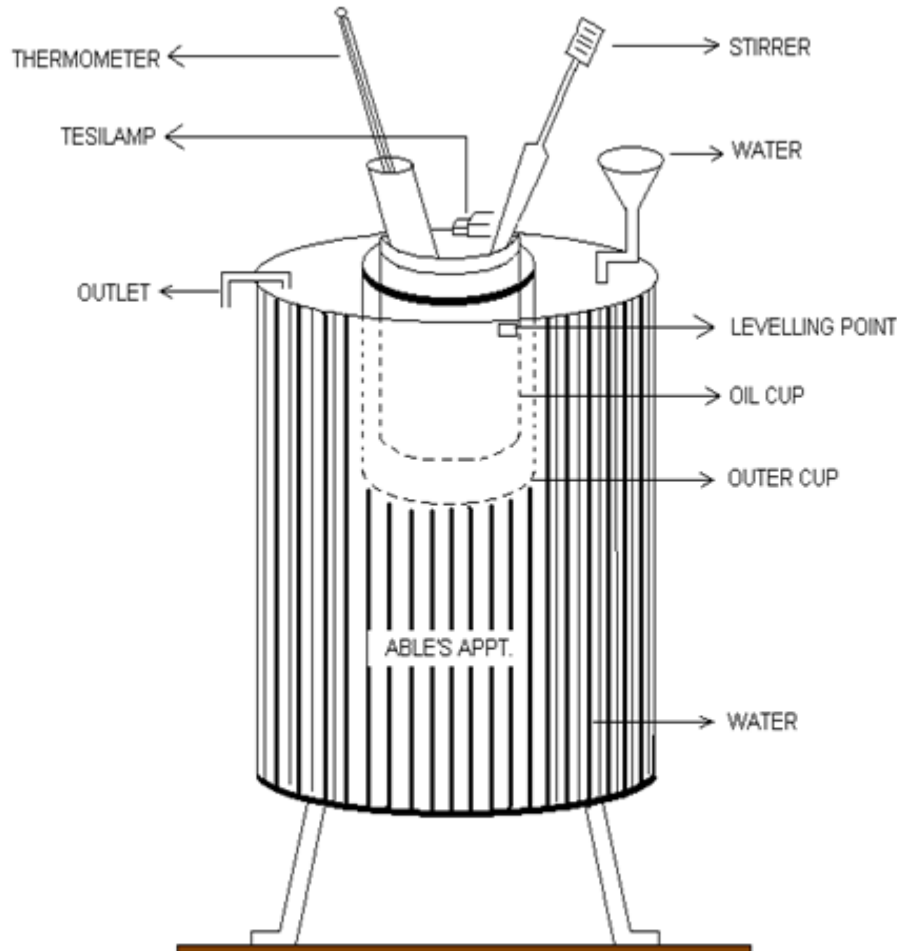**Department of Basic Sciences & Humanities**

Date of Approval: 22/07/2024   Signature of HoD   Version:3
(Dr.Mamta Takarkhade)

Experiment No: 2

Aim: To determine Flash Point of given fuel by Abel's closed cup Apparatus. (CO1)

Apparatus: Abel's Flash point Apparatus (Close cup, Air Jacket) Oil sample, Match Box, Thermometer

Diagram of Abel's flash point apparatus

ABEL'S FLASH POINT APPARATUS

Theory:

Flash point is the lowest temperature at which the oil gives off sufficient vapour to ignite momentarily when a flame of standard dimension is brought near the surface of the oil for a prescribed rate in an apparatus of specific dimension.

Procedure:

1) Fill up the oil cup with the test oil up to the mark at room temperature.

2) Insert the thermometer to check the temperature of the oil under test.

3) Oil should be heated at the rate of 3-5 ºC per minute

4) Bring the standard flame near the oil by opening the slit at the interval of 2ºC , above 30ºC. Stirring is necessary just before opening the slit (for each temperature) to observe the flash. Note down the temperature at which the first flash is observed.

5) The exact temperature of flash could be found out in decreasing order by keeping the cup on the ring outside the apparatus by applying the same method.

Significance:

1) Lubricating oil selected for the job should have a flash point, which is reasonably above its working temperature for ensuring safely against fire hazard during storage transport and use of lubricating oil. So proper lubricating oil can be selected if flash point is known.

2) The flash point of the oil determined in the standard conditions helps in selection of the oil as a lubricant. As the flash point has the standard value for pure lubricant oil. But, if it is deviating, it shows that oil is not pure is either oil adulterated or contaminated.

Observation table:

Increasing order

Sr. No	Temperature in ° C	Observation
1	30	

2	35	
3	40	
4	45	
5	45	
6	51	
7	54	
8	57	
9	60	

Decreasing order

Sr.No	Temperature in $^\circ$ C	Observation
1	60	
2	57	
3	54	
4	51	
5	48	
6	45	
7	40	
8	35	
9	30	

Result:

The Flash Point of the given oil was found out to be...……….. ^{0}C

Course Outcome:

	Course Outcome	PO
CO 1	**Interpret** the types of Energy sources and its properties and application.	PO1, PO9,PO10,PO12

Program Outcomes:

Course Outcome	Program Outcomes											
	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12
CO 1	2	-	-	-	-	-	-	-	1	1	-	2

Viva Questions:

1. What is flash point?

2. What is the significance of flash point and fire point measurement?

3. What is meant by flash point 'open' & flash point 'closed'? Which one is reproducible?

4. Mention the contaminants, which can increases, or decreases the flash point of lubricating oil?

Conclusion: Thus, we have studied the determination of flash point of lubricating oil by Abel's successfully.

Signature of Lab Course Coordinator_____

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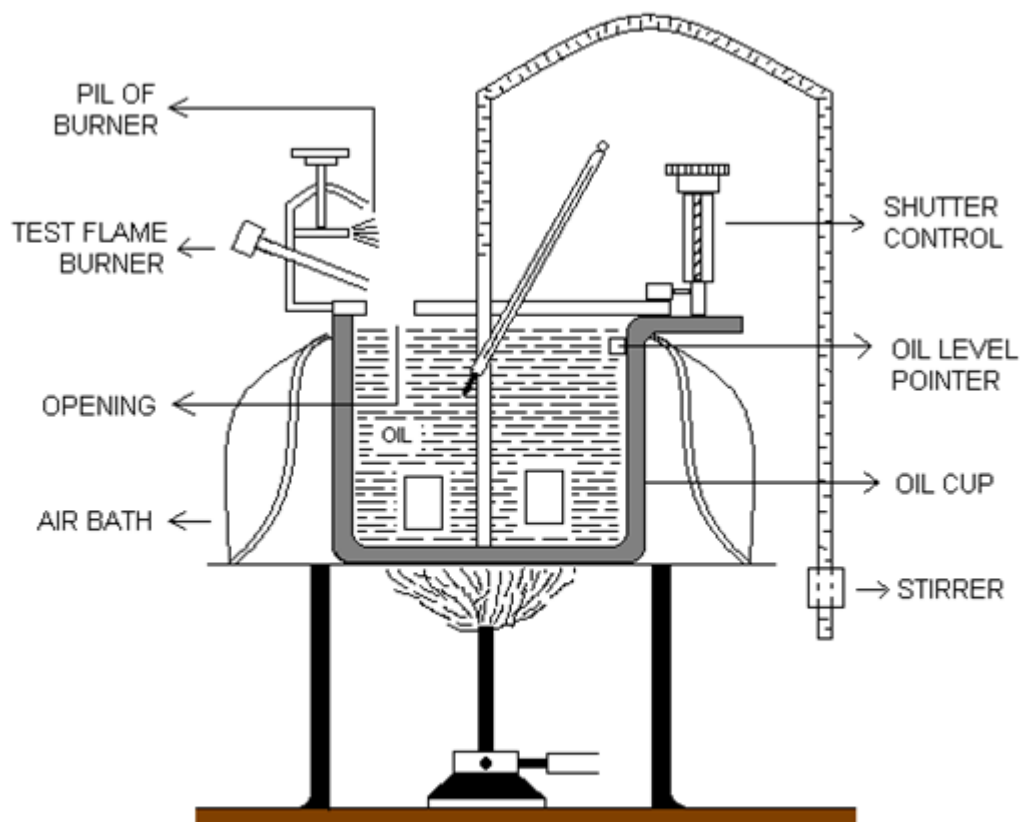**Department of Basic Sciences & Humanities**

Date_____

Aim: To determine flash point of oil by Pensky-Marten's closed –cup apparatus.[CO1]

Apparatus: Pensky- Martin's Apparatus (Closed Cup, Air Jacketed), Oil sample,

Match Box, Thermometer.

PENSKY MARTEN'S FLASH POINT APPARATUS

Theory:

"Flash point is the lowest temperature at which the oil gives off sufficient vapour to ignite momentarily when a flame of standard dimension is brought near the surface of the oil for a prescribed rate in an apparatus of specific dimension."

"Fire point is the lowest temperature at which the vapours of oil burn continuously, atleast for 5 seconds when a small flame is brought near it."

In most of cases, the fire point of an oil is about 5 to 40°C higher than its flash point.

A good lubricating oil is that which should not volatalise under the working temperature. Even if it volatlises, the vapours formed should not form a inflammable mixture with air under the

conditions of lubrication. The oil selected for a particular job should have a flash point, which is sufficiently above its working temperature. This ensures the safety against the risk of fire hazards during the storages, transport and use of the lubricating oil.

Procedure :

1) Fill up the oil cup with the test oil up to the mark at room temperature.

2) Insert the thermometer to check the temperature of the oil under test.

3) Oil should be heated at the rate of 5° C per minute.

4) Bring the standard flame near the oil by opening the slit at the interval of 5° C, above 49° C. stirring is necessary just before opening the slit (for each temperature) to observed the flash. Note down the temperature at which the first flash is observed.

5) The exact temperature of flash could be found out in decreasing order by keeping the cup on the ring outside the apparatus by applying the same method. Find out the flash at the rate of 2° C interval in decreasing order.

Significance:

3) Lubricating oil selected for the job should have a flash point, which is reasonably above its working temperature for ensuring safely against fire hazard during storage transport and use of lubricating oil. So proper lubricating oil can be selected if flash point is known.

4) The flash point of the oil determined in the standard conditions helps in selection of the oil as a lubricant. As the flash point has the standard value for pure lubricant oil. But, if it is deviating, it shows that oil is not pure is either oil adulterated or contaminated.

Observation table:

Increasing order

Sr. No	Temperature in $^\circ$ C	Observation
1	80	
2	85	
3	90	
4	95	

Sr.No	Temperature in °C	Observation
5	100	
6	105	
7	110	
8	115	
9	120	

Decreasing order

Sr.No	Temperature in ° C	Observation
1	120	
2	115	
3	110	
4	105	
5	100	
6	95	
7	90	
8	85	
9	80	

Result:

The Flash Point of the given oil was found out to be..……….. ºC

Course Outcome:

	Course Outcome	PO
CO 1	**Interpret** the types of Energy sources and its properties and application.	PO1, PO9,PO10, PO12

Program Outcomes:

Course Outcome	Program Outcomes											
	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12
CO 1	2	-	-	-	-	-	-	-	1	1	-	2

Viva Questions:

1. What is flash point?
2. Name the apparatus used to determine the flash point?
3. What is the significance of determination of flash point?
4. Why the flash point of lubricating oil should be above than the working temperature of machine?
5. What is fire point?
6. Why closed cup apparatus give more accurate result than the open cup apparatus?

Conclusion: Thus, we have studied the Flash point of lubricating oil by Pensky- Martine closed cup apparatus successfully.

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Date_____

Experiment No.3

Aim: -To determine capacity of cation exchange resin. [CO2]

Appratus: - Burette, Pipette, Column, Reservoir, beaker, Glass rod etc.

Chemials: - 0.1N NaOH ,0.1 N HCl, Phenolphthalein, Cation exchange resins, Glass wool.

Theory:- The exchange capacity of a resin is dependent upon the number of ionizable sites on the resin fame work. The ion exchange capacity of a given ion exchanger can be expressed either as mg equivalents per unit of wet volume.

Principal of finding the ion exchange capacity of cation exchanger

1. The cation exchange is first converted to its H+ form by passing 0.1 N HCl solution.

$$RSO^{3-}Na^+ \quad \underrightarrow{\quad HCl \quad} \quad RSO^{3-}H^+$$

2. The ionisable H+ ions of the resin are then exchanged for Na+ ions by passing a standard solution of NaCl.

$$RSO^{3-}H^+ \quad \underrightarrow{\quad NaCl \quad} \quad RSO^3Na^+ + HCl$$

The hydrogen ions are released in equivalent amounts of Na^+ ions. The so released HCl is titrated against standard NaOH solution. Then, the exchange capacity of resin is determine by measuring the milligram equivalents sodium ion which are absorbed by 1 gm of the resin in the H+ form.

Procedure:-   **A) Preparation of column:**

a)   The resin must be washed before use. This is done by stirring the beads into a beakerful of distilled water and allowing to settle. The supernatant liquor containing any small fragments and impurities is sucked off using a tube attached to a filter pump.

b)   A slurry of resin and water is poured into a Chromatography tube and resin beads are allowed to sediment out. Care must be taken so that air bubbles are not trapped. If necessary, use back washing to produced a satisfactory packing.

B) Regeneration of ion exchange resin

1. This can be done by washing with 10% solution of HCl.

2. Wash the resin several times with distilled water and check for the completion   of the process with pH paper.

C) Determination of exchange capacity of resin

Allow 0.1N NaCl solution to pass through the column at a rate of 2 mL/minute. Frequently Check the effluent with pH paper. When it shows no change in colour, it suggests that replacement is complete. The collection effiuent solution is then titrated against 0.1N NaOH using phenolphthalein as indictor. At the end point,the solution turns pink.

Observations: -

1. Weight of Cation exchanger resin packed in the column = Wc =_____g.

2. Normality of NaOH= 0.1 N

3. Volume of NaOH consumed = Vc = _____ml

Calculation:-

1. Ion exchange capacity of the cation exchanger = $\dfrac{Vc \times 0.1}{Wc}$ m eq/g

$$= \underline{\hspace{3cm}} \text{m eq /g}$$

Result:-The Ion exchange capacity of the cation exchanger =---------- m eq/ g

Course Outcome:

	Course Outcome	PO
CO 3	**Explain** the manufacturing of Cement, properties and different types of cement	PO1, PO2,PO9, PO10,PO12

PO Mapping:

Course Outcome	Program Outcomes											
	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12

CO 3	2	1	-	-	-	-	-	-	1	1	-	1

<u>**Viva Questions:**</u>

Q1. What is ion exchange process?

Q2. What is cation exchange resin? Explain.

Q.3. How ion exchange column are made?

Q4. What are the applications of ion exchange resin.

Conclusion: Thus, we have studied the capacity of cation exchange resin successfully.

Signature of Lab Course Coordinator_____

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Experiment No.4

Aim: -To determine Heat of Hydration of Given Material. [CO2]

Appratus: - Beakers (250 mL) : Three Beaker (500 mL) ,Thermometer (110°C) ,Glass rod. Cotton wool, Small wooden block , Small piece of cardboard Stirrer

Chemials: -0.1N NaOH, 0.1 N HCl, given material.

Theory:- In thermochemical measurements generally aqueous solutions are mixed therefore, water in the reaction medium and the temperature changes result due to the chemical reactions taking place in solution. THERMOCHEMICAL MEASUREMENT 21 According to law of conservation of energy, the sum of enthalpy changes taking place in the calorimeter (loss and gain of energy) must be zero. Thus, we can write the following equation- * Density of the solutions is 4 to 6% higher than that of pure water and heat capacity is about 4 to 8% less than pure water so the product of density and heat capacity (dC_p) is nearly the same as the product of pure water. (ΔH_1) Heat gained by calorimeter, thermometer and stirrer (ΔH_2) Enthalpy change of solution/water in calorimeter (ΔH_3) Enthalpy change of added solution/ water in calorimeter (ΔH_4) Enthalpy change of reaction + + + = 0 .. (6) • Copper sulphate/ potassium nitrate : 2g In these reactions we take the product of density and heat capacity of solutions, dC_p, to be 4.184 J.mL–1.K–1, nearly the same as that of pure water.* Solution formation often accompanies heat changes. Enthalpy of solution is the amount of heat liberated or absorbed when one mole of a solute (solid/liquid) is dissolved in such a large quantity of solvent (usually water) that further dilution does not make any heat changes

Procedure:

A) Determination of Calorimeter constant of calorimeter (Beaker)

(i) Take 100 mL of water in a 250 mL beaker marked 'A'.

(ii) Place this beaker on a wooden block kept in a larger beaker of capacity 500 mL

(iii) Pack the empty space between the large and the small beaker with cotton wool. Cover the beaker with a cardboard. Insert thermometer and stirrer in the beaker through it.

(iv) Record the temperature of water. Let this temperature be c °C.

(v) In another beaker of 250 mL capacity marked 'B' take 100 mL of hot water (50-60°C).

(vi) Note the exact temperature of hot water. Let this temperature be °C.

(vii) Lift the card board and pour the hot water contained in beaker B into beaker A. Stir the mixed water and note the temperature. Let this temperature be tm °C.

(viii) Calculate the calorimeter constant of the beaker by using the expression (5) given above. (Remember the three temperatures are in the order th > tm > t c).

B. Determination of Enthalpy of Dissolution

(i) Take 100 mL of distilled water in the beaker of which calorimeter constant has been determined and place it on a wooden block kept in a larger beaker of capacity 500 mL

(ii) Pack the empty space between the larger and the smaller beaker with cotton wool and cover with a cardboard.

(iii) Record the temperature of water already taken in the small beaker. Let this be t' 1 °C.

(iv) Add weighed amount, say W1 g of well powdered copper sulphate in water and stir the solution with a stirrer till the entire amount of copper sulphate dissolves.

Observations and Calculation:-

Temperature Measure =

Sr No	Time	Temperature
1	0 Min	
2	5 Min	
3	10 Min	
4	15 Min	
5	20 Min	
6	25 Min	
7	30 Min	
8	35 Min	

Result:- Heat of Hydration of Given Material is found to be =_____

Course Outcome:

	Course Outcome	PO
CO 3	**Explain** the manufacturing of Cement, properties and different types of cement	PO1, PO2,PO9, PO10,PO12

PO Mapping:

Course Outcome	Program Outcomes											
	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12
CO 3	2	1	-	-	-	-	-	-	1	1	-	1

Conclusion: Thus, we have studied the capacity of cation exchange resin successfully.

Signature of Lab Course Coordinator_____
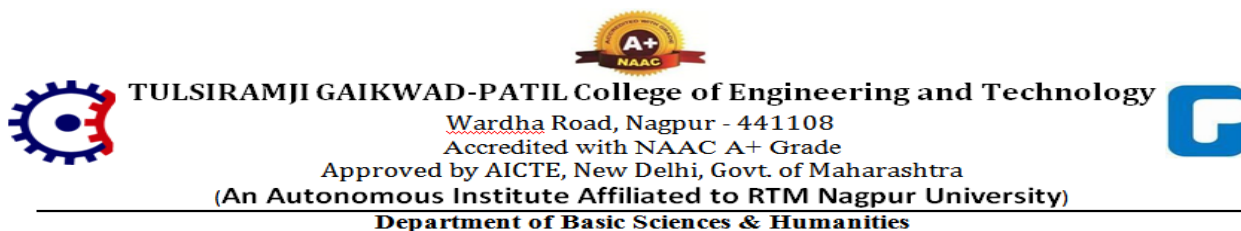
**Date_____**

Experiment No.5

Aim: To determine Temporary & Permanent hardness of water by complexometric method.[CO3]

Apparatus: Burette, Pipette, Conical flask, Beaker, measuring cylinder.

Chemicals : 0.01 M $MgSO_4$ Solution, EDTA solution, $(NH_4Cl – NH_4OH)$ buffer solution, EBT indicator.

Theory: Hardness is defined as soap consuming capacity of water. It is mainly due to the presence of Ca^{++} & Mg^{++} ions in water. Soap consists of sodium salt of long chain fatty acids

such as oleic acid, palmate acid. When adequate amount of water is treated with soap, Ca & Mg ions present in the water react with soap to form insoluble precipitate of Ca & Mg soap with no detergent value.

$$2C_{17}H_{35}COONa + MgSO_4 \rightarrow (C_{17}H_{35}COO)_2 Mg + 2Na^+$$
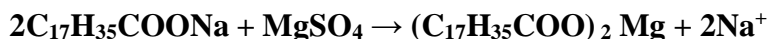
Hardness is of two types: -

1. Temporary Hardness   2. Permanent Hardness.

Temporary hardness is mainly due to bicarbonates of Ca & Mg & can be easily removed by boiling.

$$Ca(HCO_3)_2 \xrightarrow{\text{boiling}} CaCO_3 \downarrow + H_2O + CO_2\uparrow$$

$$Mg(HCO_3)_2 \xrightarrow{\text{boiling}} Mg(OH)_2 + 2CO_2\uparrow$$
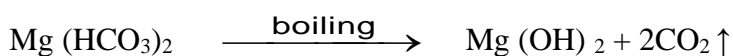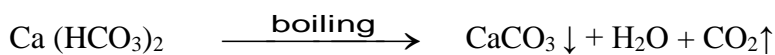
EDTA is a complexometric agent having structure
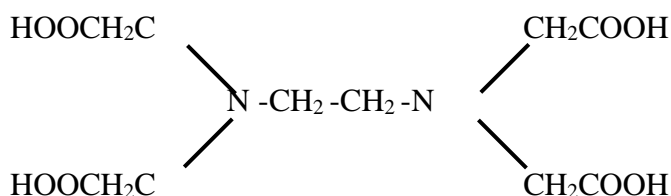
Due to its low solubility in water, we use di or tetra sodium salt of EDTA. It forms complex with Mg^{2+} & Ca^{2+}. The general formula of EDTA metal ion complex is

$M^{2+} = Ca^{2+}$ or Mg^{2+}
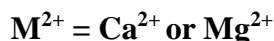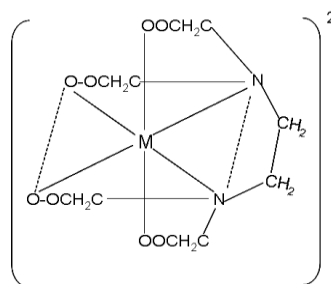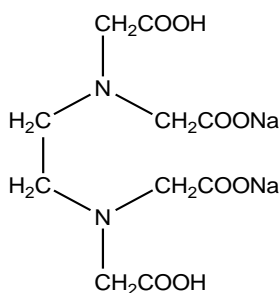
EBT is used as metal ion indicator & its structure is

EBT is sodium, 1-hydroxy, 2-napthylazo 6-nitro, 2-napthyl Sulphonate.

Metal ion react with EBT & form metal ion EBT which is unstable & wine red in colour. The metal EBT complex reacts with EDTA to form stable metal EDTA complex which is colourless and EBT is set free which is blue in colour.

**PROCEDURE: - Part [A] – Determination of Molarity of EDTA**

Pipette out 10 ml of standard 0.01 M $MgSO_4$ solution in conical flask. Add 5 ml of buffer solution and 2-3 drops of EBT indicator. Wine red colour appears. Titrate this solution with EDTA solution filled in the burette, till the colour changes from wine-red to clear blue.

Part [B] – Determination of total hardness

Pipette out 25 ml of hard water sample in conical flask. Add 5 ml of buffer solution & 2-3 drops of EBT indicator. Wine red colour appears. Titrate with EDTA solution. At the end colour changes from wine red to blue

Part [C] – Determination of permanent hardness

Take 50 ml of hard water in a beaker. Boil it till the level of water become half i.e. 25 ml. Cool it & then pour it in 100 ml Volumetric flask & mix it with distilled water to make volume upto 100 ml. Fill the burette with EDTA solution. Pipette out 25 ml hard water sample from vol^m flask in conical flask then add 5 ml buffer solution & 2-3 drops of EBT indicator wine red colour appears. Titrate with EDTA solution. At the end colour changes from wine-red to blue.
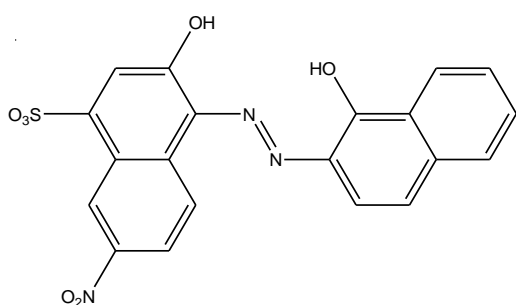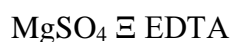

Observation:

PART [A]: Standardization of EDTA

Sr. No.	Volume of MgSO₄ taken	Volume of EDTA required	Constant Reading
1.	10 ml		
2.	10 ml		

3.	10 ml		

CALCULATION: Standardisation of EDTA

[A]   To calculate Molarity of EDTA

$MgSO_4$ Ξ EDTA

$M_1V_1 = M_2V_2$

_____ x 10 = M_2 x _____

M_2= _____M

∴ **Molarity of EDTA = _____ M**

PART [B]: Total hardness determination

Sr.No.	Volume of Hard water taken	Volume of EDTA required	Constant Reading
1.	25ml		
2.	25 ml		
3.	25 ml		

Calculation: - Total Hardness

[B] To determine total hardness of water.

   1 ml of 1 M EDTA solution = 100 mg of $CaCO_3$

____ ml of ____ M EDTA = _____ x ____ x 100 = ____ mg / 25ml

For 1000 ml, Permanent hardness = _____ x 40 = _____ppm.

PART [C]: - Permanent hardness of water

Sr.No.	Volume of Boiled Hard water taken	Volume of EDTA required	Constant Reading
1.	25ml		

2.	25 ml		
3.	25 ml		

Calculation: - Permanent Hardness

[C]   To calculate Permanent hardness of water.

1 ml of 1 M EDTA solution = 100 mg of $CaCO_3$

____ ml of ____ M of EDTA solution = 100 x ____ x ____

=_____ mg for 25 ml

For 1000 ml, Permanent hardness = _____ x 40 = _____ppm.

Calculation: - Temporary Hardness

[D]   To calculate Temporary hardness of water.

Temporary hardness = Total hardness - Permanent hardness

RESULT: 1. Total hardness of the given water sample is found to be   _____ ppm.

2. Permanent hardness of given water sample is found to be _____ ppm.

3. Temporary hardness of given water sample is found to be _____ ppm.

Course Outcome:

	Course Outcome	PO
CO 3	**Differentiate** water pollution and its softening process.	PO1, PO2,PO9, PO10,PO12

PO Mapping:

Course Outcome	Program Outcomes											
	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12
CO 3	2	1	-	-	-	-	-	-	1	1	-	1

PRECAUTIONS:

1. Apparatus should be washed carefully.
2. There should not be any air bubble in the burette.
3. Take the reading carefully.

Conclusion: Thus, we have studied the determination of Temporary and Permanant hardness by complexometric titration method successfully.

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Date_____

Experiment No.6

Aim: - **Determination of hardness of water due to Calcium and Magnesium ions separately.[CO3]**

Apparatus:- Beaker, Burette, Pipette, Conical Flask, Measuring Jar, etc.

Chemicals:- EDTA solution, Buffer solution (NH_4OH + NH_4Cl) Sodium hydroxide, EBT indicator P&R indicator.

Chemical Equations:-   M + ln $\rightarrow$ M – ln complex

              M – ln + EDTA $\rightarrow$ M – EDTA + ln

              Where M $\rightarrow$ Metal Ion

              ln $\rightarrow$ Indicator

Theory: - Ca^{2+} can be exclusively titrated in presence of Mg^{2+} with EDTA solution using Calcon indicator or less satisfactorily with Murexide indicator then the combine Ca^{2+} and Mg^{2+} can be determined by titration with EDTA solution using Eriochrome Black-T indicator. The difference between the two titer value correspondes to the Magnesium hardness.

Procedure:-

Part I: - Determination of Total Hardness

Fill the burette with EDTA solution. Pipette out 25 ml of hard water sample in a conical flask. Add 5 ml of Buffer solution and 2-3 drops of EBT indicator. Titrate with EDTA solution filled in the Burette till the colour changes from wine red to blue. Repeat the titration for constant readings.

Part II: - Determination of Calcium Hardness.

Pipette out 25 ml of hard water sample add 5 ml NaOH solution and add 2-3 drops of P&R indicator. Pink colour appears, titrate with EDTA solution filled in the Burette, till colour changes from pink to violet blue. Repeat the titration for constant readings.

Part III:- Determination of Magnesium Hardness

Magnesium hardness =Total Hardness - Calcium Hardness

Observation & Calculations

1. **Total Hardness Determination**.

Sr. No.	Volume of Hard Water Taken	Volume of EDTA required	Titrate Value
1	10 ml		
2	10 ml		
3	10 ml		

2. **Calcium Determination.**

Sr. No.	Volume of Hard Water Taken	Volume of EDTA required	Titrate Value
1	25 ml		

2	25 ml		
3	25 ml		

Calculations:

1. *To determine total hardness of water.*

    1 ml of 1M EDTA solution = 100 mg of $CaCO_3$

    _____ ml of 0.01M EDTA = _____* 0.01 * 100 mg of $CaCO_3$

    25 ml of hard water solution = _____* 0.01 * 100 mg of $CaCO_3$

    For 1 lit of hard water =_____ * 40 mg/lit.

    =- _____mg/lit.

2. *To determine Calcium hardness.*

    1 ml of 1M EDTA solution = 100mg of $CaCO_3$

    _____ ml of 0.01M EDTA = _____* 0.01 * 100 mg of $CaCO_3$

    25 ml of hard water solution = _____* 0.01 * 100 mg of $CaCO_3$

    For 1 lit of hard water =_____* 40 mg/lit

    =-_____mg/lit.

3. *To determine Magnesium hardness*

    Magnesium hardness =Total Hardness - Calcium Hardness

Result:-   1. In the given water sample total hardness of water equivalent to $CaCO_3$ is found to be _____ ppm.

2. Calcium hardness equivalent to to $CaCO_3$ is found to be_____ ppm.

3. Magnesium hardness equivalent to to $CaCO_3$ is found to be_____ ppm.

Course Outcome:

	Course Outcome	PO
CO 3	**Differentiate** water pollution and its softening process.	PO1, PO2,PO9, PO10, PO12

PO Mapping:

Course Outcome	Program Outcomes											
	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12
CO 3	2	1	-	-	-	-	-	-	1	1	-	1

Viva Questions:-

Q1.What is Hardness? Why it is due to?

Q2.Why soap is not effective in hard water?

Q3. Differentiate between temporary and permanent hardness.

Q4.What is the role of buffer solution in complexometric titration?

Q5. Write down the biological role of magnesium and calcium?

Q6. Give the structure of EDTA and EBT?

Conclusion: Thus, we have studied the hardness of water due to Calcium and Magnesium ions separately successfully.

Signature of Lab Course Coordinator_____

Date_____

Experiment No.7

Aim: To determine *p*H of waste water by using pH Meter .[CO4]

Apparatus: pH meter, Beaker, Wash bottle, Tissue Paper, etc.

Chemicals: Solution of *p*H 4.01 (Potassium Hydrogen Phthalate 0.05M), Solution of pH 7.0, Solution of pH 9.15 (Borax 0.01M), Distilled water, Saturated KCl Solution.

Theory: For many purpose, especially with small concentrations, it is cumbersome to express concentrations of hydrogen and hydroxyl ions in terms of moles per liter (mol /l). A very convenient method was proposed by S. P. L. Sorensen (1909). He introduced the hydrogen ion exponent *p*H defined by the relationships

$$pH = \log_{10}\left(\frac{1}{\left[H^+\right]}\right) = -\log_{10}\left[H^+\right] \text{ or } \left[H^+\right] = 10^{-pH}$$

The quantity *p*H is thus the logarithmic (to the base 10) of the reciprocal of the hydrogen ion concentration or the logarithm of the hydrogen ion concentration with negative sign. It is used
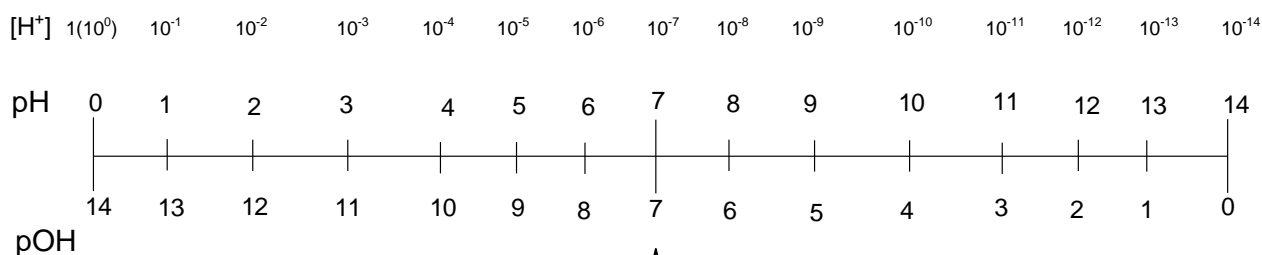
for the determination of the degree of acidity and/or alkalinity of a solution and is expressed by the *p*H scale which is a series of numbers between 0 to 14.

Significance:

1. Degree of acidity and/or alkalinity can be determined using *p*H meter.
2. *p*H meter can be used for the determination of the end-points in acidimetric and in precipitation titrations.

Procedure:

1. Switch on the instrument and allow it to warm up for some time.
2. Prepare the buffer solutions of 4.01 *p*H, 7.0 *p*H and 9.15 *p*H by dissolving the buffer tablets in specified volume of distilled water. These are necessary for calibration of the instrument.
3. If the instrument is equipped with a manual temperature control, set the control to the measured temperature of the solutions.
4. Insert the electrode assembly into the standard buffer solution (say 7.0 *p*H) taken in a clean and dry beaker.

5. Carefully adjust the "Set Buffer" control until the meter reading display the known *p*H (7.0 *p*H) of the selected buffer solution.
6. Withdraw the electrode assembly, rinse the electrode with the distilled water and insert it into a beaker containing second buffer solution (say 4.01 *p*H). If the meter reading does not agree exactly with the known *p*H of the second buffer solution (i.e. 4.01 *p*H), adjust the "Slope" control to achieve the same.
7. Repeat steps from (4) to (6) to ensure the satisfactory calibration using the third buffer solution (i.e. 9.15 *p*H).
8. Withdraw the electrode, rinse with the distilled water and introduced into the "test" (Given water samples) solution taken in another small and clean beaker. Read off *p*H of the solution, after setting the selector switch to the expected *p*H range.
9. Remove the electrode assembly out, rinse in distilled water and place it in distilled water taken in another clean beaker. Put back the selector switch to "zero position". Repeat the same procedure for every subsequent *p*H determinations.
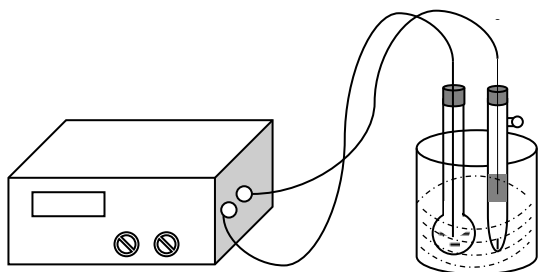
| $[H^+]$ | $1(10^0)$ | 10^{-1} | 10^{-2} | 10^{-3} | 10^{-4} | 10^{-5} | 10^{-6} | 10^{-7} | 10^{-8} | 10^{-9} | 10^{-10} | 10^{-11} | 10^{-12} | 10^{-13} | 10^{-14} |

| pH | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |

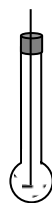| pOH | 14 | 13 | 12 | 11 | 10 | 9 | 8 | 7 | 6 | 5 | 4 | 3 | 2 | 1 | 0 |

Acid   Neutral   Alkaline

pH meter assembly Electrode
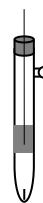
Glass electrode   Calomel

Observation Table:

Sr. No.	Samples taken	*p*H measured
1	Water Sample A	
2	Water Sample B	
3	Water Sample C	
4	Water Sample D	

Result: *p*H of given water samples was found to be,

Sample A: Sample B:

Sample C: Sample D:

Course Outcome:

	Course Outcome	PO
CO 4	**Illustrate** bulk properties and processes used in thermodynamics, Different types and application of batteries	PO1, PO2, PO6, PO7, PO9, PO10, PO12

PO Mapping:

Course Outcome	Program Outcomes											
	PO1	**PO2**	**PO3**	**PO4**	**PO5**	**PO6**	**PO7**	**PO8**	**PO9**	**PO10**	**PO11**	**PO12**
CO 4	2	1	-	-	-	1	1	-	1	1	-	1

<u>Viva Questions:</u>

Q1. Explain pH,pH-scale,pKa,pKb

Q2. What are potentiometric titrations?

Q3. Which electrode is used in pH titrations?

Q4. Explain Buffer solution.

Conclusion: Thus, we have studied the *p*H of waste water successfully.

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Date_____

Experiment No.8

Aim: To determine Electrode Potential of Copper metal by Galvanic cell . **[CO4]**

Apparatus-Beaker, Salt Bridge, zinc rod and copper rod Electrode

Chemicals:1.0M Zinc sulphate solution : 40mL 0.25 M, 0.1M, 0.05M, 0.025 M and 0.0125M
Copper sulphate solutions.

Theory:

Due to reaction between metal and solution an electrode double layer is form around metal. It gives rise potential difference between metal and solution known as electrode potential. Electrode potential is measure tendency of metal electrode to lose or gain electron when it is in contact with a solution of its own salt solution.
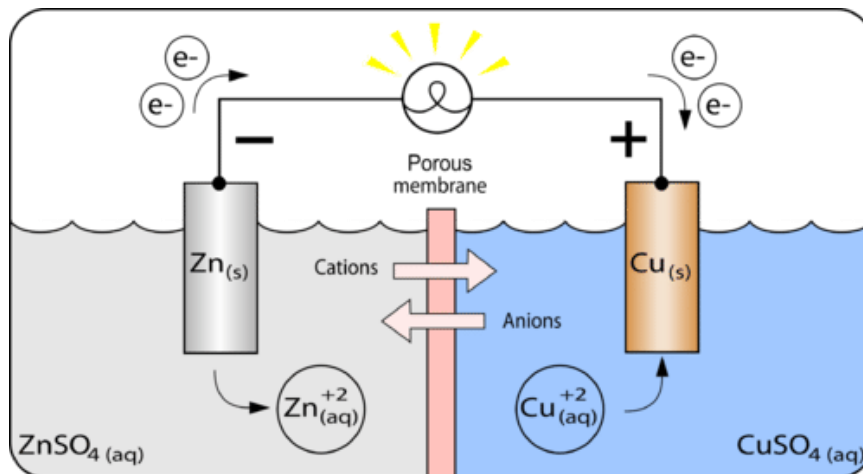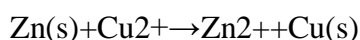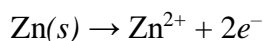
Cell description conventions:

In order to make it easier to describe a given electrochemical cell, a special symbolic notation has been adopted. In this notation the cell we described above would be,
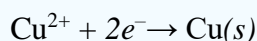
Zn*(s)* | Zn^{2+}*(aq)* || Cu^{2+}*(aq)* | Cu*(s)*

Reaction:

Zn(s)+Cu2+→Zn2++Cu(s)
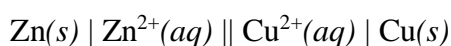
| *Left electrode:* | Zn*(s)* → Zn^{2+} + 2e$^-$ | *oxidation* |
| *Right electrode:* | Cu^{2+} + 2e$^-$→ Cu*(s)* | *reduction* |

Procedure:

1. Make surface of zinc rod and copper rod smooth by using polish paper then clean with dil. HCl and water

2. Take 1 M ZnSO$_4$ and 1M CuSO$_4$ solution in two different beakers.

3. Place Zn rod in ZnSO4 and copper rod in CuSO4.

4. Connect Zn rod to negative terminal and copper rod to positive terminal of digital multimeter.

5. Place salt bridge in both solutions.

6. Note down the cell EMF in volts displayed by digital multimeter.

7. Calculate electrode potential of copper as per given calculation.

Observation:-

1. Temperature=….

2. Theoretical value of reduction potential of Zn=-0.76

3. Electrode potential of cell=…….V

Result:- The Electrode Potential of Copper metal by Galvanic Cell was found to be…………….V

<u>**Viva Questions:**</u>

(i)   For the reaction given below, apply Le-Chatelier principle to justify the results recorded by you and also bring out mathematical rationalisation of your results. $Zn(s) + Cu^{2+} (aq)$ Zn^{2+} $(aq) + Cu(s)$,

(ii) Determine the slope of the graph. Match experimental value with the theoretical value. On what factors does the value of slope depend?

(iii) Devise another experiment to study the variation in cell potential with concentration of one of the ions involved in a cell reaction.

(iv) What factor is kept in mind while selecting an electrolytic solution for the construction of a salt bridge?

(v) Is it possible to measure the single electrode potential?

Course Outcome:

	Course Outcome	PO
CO 4	**Illustrate** bulk properties and processes used in thermodynamics, Different types and	PO1, PO2,PO6, PO7, PO9, PO10, PO12

application of batteries

Course	Program Outcomes											
Outcome	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12
CO 4	2	1	-	-	-	1	1	-	1	1	-	1

Conclusion: Thus, we have studied the electrode potential determination of copper metal by Galvanic cell successfully.

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Date_____

Experiment No.9

Aim: -To estimate the amount of Zinc Deposited During Electroplating. [CO5]

Appratus: - Zinc plate : Copper plate , Beaker (50 mL) ,Voltmeter (Potentiometer), Salt bridge.

Chemials: -1.0M Zinc sulphate solution : 40mL 0.25 M, 0.1M, 0.05M, 0.025 M and 0.0125M Copper sulphate solutions.

Theory:- The cell under investigation in this experiment is represented as follows: Zn(s)/Zn 2+(aq., 1.0M) ||Cu 2+ (aq., x M)/Cu(s) Here x M denotes varying concentrations of Cu2+(aq) ions. In other words, to study the variation in cell potential with concentration, the concentration of
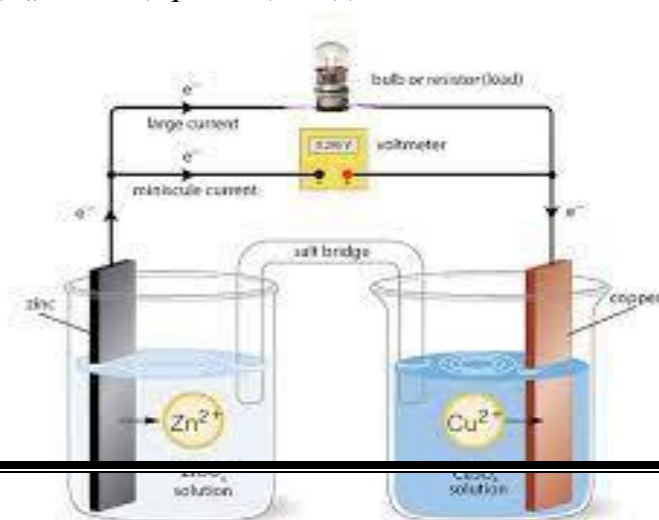
Cu2+ (aq.) is varied while that of Zn2+ (aq) is kept constant. The measured cell potential enables us to calculate the electrode potential of Cu2+/Cu electrode for each concentration of copper (II) ions. This variation is theoretically depicted according to the equation: $E_{Cu^{2+}/Cu} = E^{\circ}_{Cu^{2+}/Cu} + \frac{0.059}{2}\log[Cu^{2+}]$ V (1) The variation in the electrode potential of Cu2+/Cu electrode consequently brings variation in the cell potential according to the relation: $E_{cell} = E_{Cu^{2+}/Cu} + E_{Zn/Zn^{2+}}$ V (2) Equation (2) clearly suggests that even if $E_{Zn/Zn^{2+}}$ V is kept constant, the variation in $E_{Cu^{2+}/Cu}$ would bring corresponding variation in Ecell (cell potential). Similarly, keeping the concentration of Cu2+ ions constant, one can study the variations in the cell potential with the variation in concentration of Zn2+ ions.

Procedure:

i) Set up the cell as given in Fig. 4.1, using 1.0 M ZnSO4 and 0.2 M CuSO4 solution.

(ii) Measure the potential difference of the cell and also keep record of the polarity of the electrodes (this will enable us to give a sign to the cell potential ECell).

(iii) Remove the salt bridge as soon as the cell potential measurement is over.

(iv) Replace the beaker of 0.2 M CuSO4 with 0.1 M CuSO4 solution in the beaker.

(v) Place the salt bridge in position and note the cell potential.

vi) Repeat this procedure for other solutions of copper sulphate in decreasing order of concentrations of copper sulphate solution.

(vii) Calculate log [Cu2+(aq)] and then $E_{Cu^{2+}/Cu}$ for each variation in the concentration of copper (II) in the solution.

(viii) Record electrode potential values of Cu2+(aq)/Cu(s) electrode for different concentrations of Cu2+ ions as given in Table 4.1.

(ix)Plot a graph for the variation of cell potential with concentration taking ($E_{Cu^{2+}/Cu}$) on y-axis and log [Cu2+(aq)] on x-axis.

Observations: -

Sr No	[Cu2+(aq)]/mol L–1	log [Cu2+(aq)]/mol L–1	Ecell/V E(Cu2+/Cu)	Experimental value
1				
2				
3				
4				
5				

Calculation:-

Result:- Amount of Zinc Deposited During Electroplating is found to be =_____

<u>Viva Questions:</u>

    (ii)   For the reaction given below, apply Le-Chatelier principle to justify the results recorded by you and also bring out mathematical rationalisation of your results. $Zn(s) + Cu^{2+}(aq)$ $Zn^{2+}(aq) + Cu(s)$,

    (iii)   (ii) Determine the slope of the graph. Match experimental value with the theoretical value. On what factors does the value of slope depend?

    (iv)   (iii) Devise another experiment to study the variation in cell potential with concentration of one of the ions involved in a cell reaction.

    (v)   (iv) What factor is kept in mind while selecting an electrolytic solution for the construction of a salt bridge?

    (vi)   (v) Is it possible to measure the single electrode potential?

Course Outcome:

	Course Outcome	**PO**
CO 4	**Predict** the causes of corrosion, its consequences and methods to minimize corrosion.	PO1, PO2,PO6, PO7, PO9, PO10, PO12

Course	Program Outcomes											
Outcome	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12
CO 4	2	1	-	-	-	1	1	-	1	1	-	1

Conclusion: Thus, we have studied the amount of zinc deposited during electroplating successfully.

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Experiment No.10

Aim: To predict the effect of temperature on rate of corrosion.[CO5]

Apparatus: Beaker (250ml), pair of tongs, electronic balance(L.C.= 0.001mg), water bath(with temperature controller),thermometer, electric oven.

Chemicals: HCl, H_2SO_4, HNO_3, NaOH.

Theory: When metal comes in the contact with atmospheric gases or liquid medium, it undergoes decay and destruction. Moisture and impurities present in the surrounding environment affects the rate of corrosion. Depending on surrounding medium corrosion are of two types. Viz, atmospheric corrosion and immersed corrosion.

Procedure:
1. Immerse accurately weighted aluminium strips in the given acids/ base at room temperature for 5-6 minutes.
2. Wash it.dry it.and weigh Al strip accurately on electronic balance.

3. Take acid/base prepared from exp.no. 4 and keep on water bath.

4. Adjust temperature of water bath at required temp.(50^0C)

5. Dip the weighed Al strip in acid/ base and wait for 5-6 min.

6. Remove the strip using pair of tongs.

7. Wash it.dry it and weigh Al strips accurately on electronic balance.

8. Find decrease in weight of Al strips.

Observations and calculations:

A) For loss in weight at room temperature = ………………………^{0}C.

Sr.No.	Solution taken	Weight of strip in mg		Change in weight of strip in mg $W_3 = W_1 - W_2$
		Before dipping W_1	After dipping W_2	
1.	HCl			
2.	H_2SO_4			
3.	HNO_3			
4.	NaOH			

B) For loss in weight at increased temperature = ………………………^{0}C.

Sr.No.	Solution taken	Weight of strip in mg		Change in weight of strip in mg $W_6 = W_4 - W_5$
		Before dipping W_4	After dipping W_5	
1.	HCl			
2.	H_2SO_4			
3.	HNO_3			
4.	NaOH			

Results :

1. Change in weight of aluminium in HCl at room temp……………..g. and change in weight of Al in HCl at temp. ……………………^{0}C Is …………………g

2. Change in weight of Al in sulphuric acid at room temp. ……………….g and change in weight of Al in sulphuric acid at temp……………..^{0}C Is ……g

3. Change in weight of aluminium in nitric acid at room temp…………g.

4. Change in weight of aluminium in nitric acid at temp…………^{0}C is………….g

5. Change in weight of Al in NaOH is at room temp………g. And change in weight of Al in sodium hydroxide at temp………^{0}C is ………….g.

Course Outcome:

	Course Outcome	PO
CO 4	**Predict** the causes of corrosion, its consequences and methods to minimize corrosion.	PO1, PO2,PO6, PO7, PO9, PO10, PO12

PO Mapping:

Course Outcome	Program Outcomes											
	PO1	PO2	PO3	PO4	PO5	PO6	PO7	PO8	PO9	PO10	PO11	PO12
CO 4	2	1	-	-	-	1	1	-	1	1	-	1

Conclusion: Thus, we have studied the rate of corrosion successfully.

Signature of Lab Course Coordinator_____