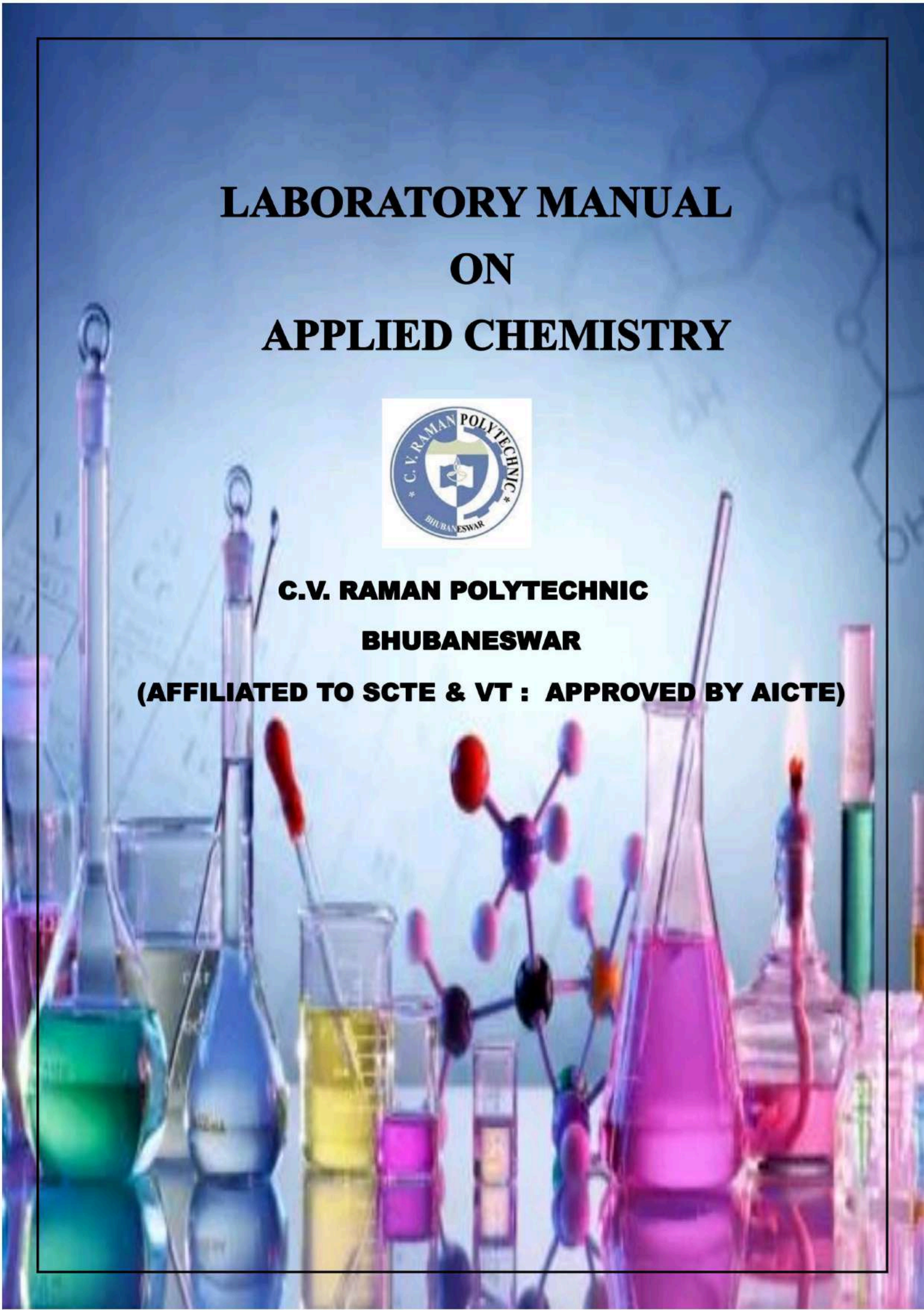

LABORATORY MANUAL
ON
APPLIED CHEMISTRY

C.V. RAMAN POLYTECHNIC

BHUBANESWAR

(AFFILIATED TO SCTE & VT : APPROVED BY AICTE)

CHEMISTRY LABORATORY MANUAL

FOR 1^{ST} /2^{ND} SEMESTER DIPLOMA COURSE

NAME : ______________________________
BRANCH : ______________________________
SCTE AND VT REGD. NO : ______________________________
SEMESTER : ______________________________
SECTION : ______________________________
GROUP : ______________________________

DEPARTMENT OF CHEMISTRY

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ENGINEERING CHEMISTRY LAB

CONTENTS

PREPARATION OF STANDARD SOLUTION

EXPERIMENT NO.01

1.0 AIM OF THE EXPERIMENT

To Prepare the standard solution of oxalic acid.

2.0 EXPERIMENTAL SET UP

2.1 LIST OF APPARATUS REQUIRED:-

- Electronic Digital/ Analytical Balance (0.0001 mg sensitivity)
- Watch glass
- Standard volumetric flask (100 ml)
- Funnel
- Beakers

2.2 LIST OF CHEMICALS REQUIRED:-

- Oxalic acid

3.0 THEORY

NORMAL SOLUTION

The normal solution is prepared by dissolving 1 g equivalent weight of solute, making 1000ml of solution.

Preparation of 0.01N of Oxalic acid

Molecular weight of oxalic acid = 126

$$\text{Equivalent weight} = \frac{\text{Molecular weight}}{\text{Basicity No}} = \frac{126}{2} = 63 \text{ (Valency of oxalic acid} = 2)$$

1g equivalent weight of oxalic acid dissolved in 1000 ml distilled water, form 1N solution.

63g of Oxalic Acid dissolved in 1000 ml distilled water, form, 1N Oxalic acid solution.

0.63g of Oxalic acid dissolved in 1000mL distilled water, form, 0.01 N Oxalic acid solution.

MOLAR SOLUTION

Number of moles of solute dissolved in 1000ml water, Forms molar solution.

Preparation of 0.02 M Oxalic Acid Solution

Molecular formula of oxalic acid —($C_2H_6O_6$)

Molecular weight = [(2×12)+(6×1)+(6×16)]=[24 + 6 + 96]=126g/mol

Weight of Oxalic Acid = Molarity x Molecular Mass of Oxalic acid×Volume

$$= \frac{1}{50} \times 126 \times \frac{100}{1000}$$

Weight of oxalic acid =0.252 g

For the preparation of 0.02 M oxalic acid solution, 0.252 g of oxalic acid is dissolved in 100ml water.

4.0 PROCEDURE

PREPARATION OF 0.01N OXALIC ACID SOLUTION

1. Clean the watch glass and weigh 0.63g of oxalic acid, and transfer it into a standard volumetric flask and add distilled water up to 1000ml mark.
2. Toggle the solution 3-4 times so that we can get a homogenous mixture of oxalic acid.

PREPARATION OF 0.02M OXALIC ACID SOLUTION

1. Clean the watch glass and weigh 0.252 g of oxalic acid, and transfer it into a standard volumetric flask and add distilled water up to 100ml mark.
2. Toggle the solution 3-4 times so that we can get a homogenous mixture of oxalic acid.

5.0 OBSERVATIONS

PREPARATION OF OXALIC ACID SOLUTION

I) Preparation of……………..M oxalic Acid Solution

For the preparation of . . . . . .M oxalic acid, Weight of oxalic acid taken =……………….

The volume of the used standard volumetric flask =……….

Il) Preparation of……………..N oxalic acid solution

For the preparation of…………N oxalic acid, the weight of oxalic acid taken =……….

6.0 RESULT

………g of oxalic acid is used for the preparation of…………..solution.

……….g of oxalic acid is used for the preparation of…………..solution.

7.0 CONCLUSION

………………………………………………………………………………

PRACTICAL RELATED QUESTION

1. **What is the formula of oxalic acid?**
2. **What is the basicity of oxalic acid?**
3. **What do you mean by the basicity of an acid?**
4. **Is oxalic acid a strong acid?**
5. **What do you mean by a molar solution**
6. **What do you mean by a normal solution**
7. **What is the standard solution?**

Student's signature

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EXPERIMENT NO.2

1.0 AIM OF THE EXPERIMENT

Prepare the standard solution of potassium permanganate

2.0 EXPERIMENTAL SET UP

2.1 LIST OF APPARATUS REQUIRED:-

- Electronic Digital/ Analytical Balance (0.0001 mg sensitivity)
- Watch glass
- Standard volumetric flask (100 ml)
- Funnel
- Beaker

2.2 LIST OF CHEMICALS REQUIRED:-

- Potassium permanganate ($KMnO_4$)

3.0 THEORY

NORMAL SOLUTION

PREPARATION OF 0.01N $KMNO_4$ SOLUTION

$$\text{Equivalent weight}=\frac{Molecular\ weight}{valency(basicity\ no)}=\frac{158}{5}=31.6gm$$

(Valency of $KMnO_4$ = 5)

31.6 g of KMn04 dissolved in 1000ml distilled water to form 1 N $KMnO_4$ solution.
0.316 g KMn04 dissolved in 1000 ml distilled water to form 0.01 N $KMnO_4$ solution.

MOLAR SOLUTION

PREPARATION OF 0.02 M $KMNO_4$ SOLUTION

Molecular weight of KMn04 = [39+55+(16 x 4)] =158
Weight of $KMn0_4$ =Molarity x Molecular Weight of KMn04 x Volume

$$=\frac{1}{50}\times 158\times\frac{100}{1000}=0.31\text{g}$$

For Preparation of 0.02 M $KMnO_4$Solution, 0.316g $KMnO_4$ is dissolved in 100 ml water.

4.0 PROCEDURE

PREPARATION OF 0.01N $KMNO_4$ SOLUTION

1. Clean the watch glass and weigh out 0.316 g of a gram of $KMn0_4$ and transfer it into a standard volumetric flask and add distilled water up to 1000ml mark.
2. Toggle the solution 3-4 times so that we can get a homogenous mixture of $KMnO_4$.

PREPARATION OF 0.02M $KMNO_4$ SOLUTION

1. Clean the watch glass and weigh out 0.316 g of a gram of $KMnO_4$ and transfer it into a standard volumetric flask and add distilled water up to 100ml mark.
2. Toggle the solution 3-4 times so that we can get a homogenous mixture of $KMnO_4$.

5.0 OBSERVATIONS

Preparation of KMn04 Solution

I) Preparation of................M $KMnO_4$ Solution

For the preparation of...............M $KMnO_4$, Weight of KMn04 taken =..........

The volume of the used standard volumetric flask =..........

Il) Preparation of................N $KMnO_4$ solution

For the preparation of..........N $KMnO_4$, the weight of $KMnO_4$ taken =..........

6.0 RESULT

.........g of oxalic acid is used for the preparation of.............solution.

.........g of oxalic acid is used for the preparation of.............solution

7.0 CONCLUSION

....

PRACTICAL RELATED QUESTION

1. **What is the formula of Potassium Permanganate ?**

2. **What is the basicity of Potassium Permanganate ?**

3. **Why is the standard solution always prepared in a volumetric flask?**

4. **Why should weights never be touched by hands?**

5. **Why is distilled water always used to prepare the standard solution?**

Student's signature

Roll no……………………

ACID- BASE TITRATION

EXPERIMENT NO.03

1.0 AIM OF THE EXPERIMENT

To Determine the strength of given sodium hydroxide solution by titrating against standard oxalic acid solution using phenolphthalein indicator.

2.0 EXPERIMENTAL SET UP

2.1 LIST OF APPARATUS REQUIRED:-

- Beaker (250mL and 100mL)
- conical flask (100mL)
- burette (50mL)
- burette stand
- pipette (10mL)

2.2 LIST OF CHEMICALS REQUIRED:-

- Oxalic acid
- sodium hydroxide
- Phenolphthalein

3.0 THEORY

ACID BASE TITRATION

Acid base titrations are based on the principles of acidimetry and alkalimetry. The chemical reaction involved in acid-base titration is known as neutralisation reaction. It involves the combination of H30+ ions with OH^- ions to form water. In acid-base titrations, solutions of alkali are titrated against standard acid solutions. The estimation of an alkali solution using a standard acid solution is called acidimetry. Similarly, the estimation of an acid solution using a standard alkali solution is called alkalimetry.

Titration

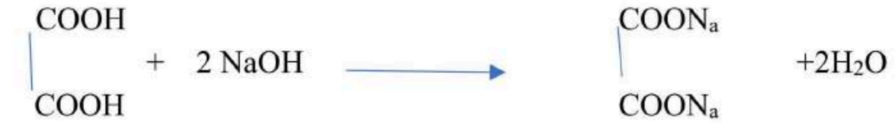
The standard solution is usually added from a graduated vessel called a burette. The process of adding standard solution until the reaction is just complete is termed as titration and the substance to be determined is said to be titrated.

Indicator

It is a chemical reagent used to recognize the attainment of end point in a titration. After the reaction between the substance and the standard solution is complete, the indicator should give a clear colour change.

END POINT AND EQUIVALENT POINT

For a reaction, a stage which shows the completion of a particular reaction is known as end point. Equivalence point is a stage in which the amount of reagent added is exactly and stoichiometrically equivalent to the amount of the reacting substance in the titrated solution. The end point is detected by some physical change produced by the solution, by itself or more usually by the addition of an auxiliary reagent known as an 'indicator'. The end point and the equivalence point may not be identical. End point is usually detected only after adding a slight excess of the titrant. In many cases, the difference between these two will fall within the experimental error. The reaction applicable in this practical is



$$\begin{array}{l} COOH \\ | \\ COOH \end{array} + 2\,NaOH \longrightarrow \begin{array}{l} COON_a \\ | \\ COON_a \end{array} + 2H_2O$$

Oxalic Acid Sodium Hydroxide Sodium Oxalate Water

4.0 PROCEDURE

1. Prepare 0.1 N NaOH solution
   (40g of NaOH dissolved in 1000mL distilled water to form 1M NaOH solution,
   4.0 g of NaOH dissolved in 1000mL distilled water to form 0.1M NaOH solution)
2. Rinse and fill given sodium hydroxide in clean burette upto zero mark.
3. Pipette out 20mL of oxalic acid and transfer it into the conical flask.
4. Add 2-3 drops of phenolphthalein indicator into the conical flask.
5. Add dropwise NaOH from burette into the conical flask and stir the mixture.
6. Repeat the step (4) until colour changes from colourless to permanent light pink. i.e. at the endpoint of titration.
7. After the formation of permanent light pink colour stop the addition and record the burette reading.
8. Repeat procedure for three consecutive readings.

5.0 OBSERVATIONS

1. The solution in burette.................................
2. Solution by pipette..
3. Indicator used...
4. End point..

6.0 OBSERVATION TABLE

Burette Readings	Pilot Reading	1	2	3	Volume of Base (VB) = Mean Difference = (1+2+ 3) / 3
Initial Burette Reading (IBR)					
Final Burette Reading (FBR)					
Difference (FBR-IBR)					

7.0 CALCULATIONS

Molarity of NaOH

NaOH vs Oxalic Acid

Molarity of Base (MB) x Volume of Base (VB) = Molarity of Acid(MA) x Volume of Acid (VA)

Molarity of NaOH = $M_B = \frac{M_A \times V_A}{V_B} = \frac{0.1 \times 20}{From\ observation\ table(V_B)}$=……………………. M

Strength of NaOH = Molarity of NaOH × Equivalent Weight of NaOH

= ……….x 40 g/L= ………………. g/L

6.0 RESULT

1. Molarity of NaOH =……….
2. Strength of NaOH -……….

7.0 CONCLUSION

……….amount of NaOH dissolve for the preparation of……...............M solution

PRACTICAL RELATED QUESTION

1. **What is titration?**

2. **What is an indicator?**

3. **What is the endpoint?**

4. **Why are the burette and the pipette rinsed with the solution with which these are filled?**

5. **Which indicator is used in the titration of oxalic acid vs sodium hydroxide? Can the titration be performed by using some other indicator?**

6. **Why should one read the lower meniscus in the case of colourless and transparent solutions and the upper meniscus for solutions of dark colour?**

7. **What is meant by the term, ‘concordant readings’?**

8. **What is acidimetry?**

9. What is alkalimetry?

10. Name a few common indicators?

Student's signature

Roll no……………………

HARDNESS OF WATER

EXPERIMENT NO.04

1.0 AIM OF THE EXPERIMENT

To Estimate the total hardness of given water sample using standard ethylene diamine tetra acetic acid (EDTA) solution

2.0 EXPERIMENTAL SET UP

LIST OF APPARATUS REQUIRED:-

- Conical flask (100 ml)
- Burette (50 ml)
- Pipette (10 ml)
- beaker (100 ml, 250 ml)

LIST OF CHEMICALS REQUIRED:-

- Water sample
- EDTA (0.1M)
- Buffer solution (pH=10)
- Eriochrome Black T(EBT) indicator

3.0 PROCEDURE

PART A:-TOTAL HARDNESS OF WATER

1. Rinse and fill the burette with 0.01M EDTA solution.
2. Pipette out 10ml of water sample in conical flask.
3. Add 5ml of buffer solution (pH= 10) and 2-3 drops of EBT indicator in the conical flask.
4. Due to addition of EBT, mixture in conical flask shows wine red colouration.
5. Titrate the solution in conical flask with EDTA till colour changes from wine red to light blue.
6. Repeat the procedure for three consequent burette reading.

PART B:-PERMANENT HARDNESS OF WATER

1. Boil the given sample for 8-10 minutes and filter.
2. Pipette out 10 mL of filtered water in conical flask.
3. Follow the procedure as per part A

4.0 OBSERVATIONS

1. Solution in burette:…………………………
2. Solution in conical flask……………………
3. Indicator……………………………………….
4. End Point ……………………………………..

5.0 OBSERVATION TABLE

PART A:-TOTAL HARDNESS OF WATER

Readings	Pilot Reading	1	2	3	= Mean Difference = (1+2+ 3) / 3
Initial Burette Reading (IBR)					
Final Burette Reading (FBR)					
Difference (FBR-IBR)					

PART B:-PERMANENT HARDNESS OF WATER

Readings	Pilot Reading	1	2	3	PH = Mean Difference = (1+2+ 3) / 3
Initial Burette Reading (IBR)					
Final Burette Reading (FBR)					
Difference (FBR-IBR)					

6.0 CALCULATIONS

PART A:-TOTAL HARDNESS OF WATER

Step 1 :

1000mL of 1M EDTA $\equiv$ 100 g $CaCO_3$

(TH) mL of 0.01M EDTA $\equiv \frac{100 \times 0.01 \times TH}{1000}$ g of $CaCO_3$=0.001$\times TH$ g of $CaCO_3$

$\equiv$ 0.001$\times TH$=0.001×………………….=…………………….(y)g of $CaCO_3$

Step 2:

10m1 of water sample contains (y) g of $CaCO_3$

1000ml of water sample contains = 100× (y)g of $CaCO_3$=…………g of $CaCO_3$

Conversion of g to mg

1000mL of water sample contains = 100× (y) g of $CaCO_3$× 1000 mg of $CaCO_3$

1000mL of water sample contains =…………………… mg of $CaCO_3$ (I)

PART B:-PERMANENT HARDNESS (PH) OF WATER

Step 1 :

IOOOmL of 1M EDTA = 100 g $CaCO_3$

(PH)mL of 0.01M EDTA $\equiv \frac{100\times0.01\times PH}{100}$g of $CaCO_3$= 0.001 × PH g of $CaCO_3$

$\equiv$0.001×PH =0.001×………………=………………(z)g of $CaCO_3$

Step 2 :

10 mL of water sample contains (z) g of $CaCO_3$

1000 mL of water sample contains = 100 × (z)g of $CaCO_3$=…………g of $CaCO_3$

Conversion of g to mg

1000 mL of water sample contains = 100× (z) g of $CaCO_3$× 1000mg of $CaCO_3$

1000 mL of water sample contains =……………………mg of $CaCO_3$ - (II)

Temporary hardness = Total Hardness - permanent hardness of water in mg of $CaCO_3$

= I-II = ………

= ……………………mg of $CaCO_3$

7.0 RESULT

1. Total Hardness for given sample of water is =…………………ppm of $CaCO_3$ equivalent
2. Permanent Hardness for given sample of water is =…………ppm of $CaCO_3$ equivalent
3. Temporary Hardness for given sample of water is =…………ppm of $CaCO_3$ equivalent

8.0 CONCLUSION

…………………………………………………………………..

PRACTICAL RELATED QUESTION

Q1: What is hardness of water?

Q2: What are the types of hardness of water?

Q3: How is hardness of water measured?

Q4: What is the cause of temporary hardness of water?

Q5: How can temporary hardness be removed?

Q6: What is the cause of permanent hardness of water?

.

Q7: How can permanent hardness be removed?

Q8: What are the effects of hard water on daily life?

Q9: Can hard water be used for drinking purposes?

Q10: How does hardness of water vary in different regions?

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Roll no……………………

ALKALINITY OF WATER

EXPERIMENT NO.05

1.0 AIM OF THE EXPERIMENT

To Determine the alkalinity of a given water sample using 0.01M sulphuric acid.

2.0 EXPERIMENTAL SET UP

2.1 LIST OF APPARATUS REQUIRED:-

- Burette
- pipette,
- conical flask (250mL)
- standard flask
- measuring cylinder

2.2 LIST OF CHEMICALS REQUIRED:-

- Standard sulphuric acid
- phenolphthalein indicator
- methyl orange
- ethyl alcohol
- distilled water.

3.0 PROCEDURE

3.1 PREPARATION OF 0.01 N SULPHURIC ACID SOLUTION

1. Take approximately 500ml distilled water in a 1000 mL standard flask.
2. Measure 200mL of 0. IN sulphuric acid and add slowly along the side of the standard flask.
3. Then dilute up to 1000ml using distilled water hence we get 0.02N sulphuric acid.

3.2 PREPARATION OF PHENOLPHTHALEIN INDICATOR

1. Weigh out l g of phenolphthalein powder and add 100 mL of ethyl alcohol or water.
2. Use a ready-made phenolphthalein indicator.

3.3 TITRATION PROCEDURE

1. Rinse and fill the 0.01 N sulphuric acid into the burette.
2. Measure exactly 100mL of sample and transfer it into 250mL conical flask. iii. Add few drops of phenolphthalein indicator into the conical flask which shows pink colouration which indicates the presence of alkalinity due to hydroxyl ions.

3. Titrate this against 0.01 N Sulphuric acid till the pink colour disappears which shows neutralization of hydroxyl ions. Record this burette reading which is accountable for phenolphthalein alkalinity.
4. To the same colourless solution in the conical flask add few drops of methyl orange indicator. The colour of the solution changes to yellow which is due to the presence of $C03^{2-}$ and $HC03^{-}$ ions in the water sample.
5. Continue the titration from the point where stopped for the phenolphthalein alkalinity.
6. Titrate till the solution becomes reddish orange.
7. The entire volume V2 of sulphuric acid is noted down and it is accountable for calculating methyl orange alkalinity.
8. Repeat the procedure for consecutive readings.

4.0 OBSERVATION

The solution in burette……………………..
Indicator…………………………………….........
End point……………………………………………

5.0 OBSERVATION TABLE

TABLE 1: PHENOLPHTHALEIN ALKALINITY

Readings	Pilot Reading	1	2	3	Difference = (1+2+ 3) / 3
Initial Burette Reading (IBR)					
Final Burette Reading (FBR)					
Difference (FBR-IBR)					

TABLE 2: TOTAL ALKALINITY

Readings	Pilot Reading	1	2	3	M=Mean Difference = (1+2+ 3) / 3
Initial Burette Reading (IBR)					
Final Burette Reading (FBR)					
Difference (FBR-IBR)					

6.0 CALCULATIONS

(i) Alkalinity due to OH^- (if P=M)

Step 1 :

1000 mL of 1N H_2SO_4 = 17 g OH^-

M mL of 0.01N H_2SO_4= $\frac{17\times0.01\times M}{1000}$g of OH^- =$\frac{17\times0.01\times......}{1000}$=(x)g of OH^-

Step 2 :

100mL of water sample contains =(x) g =…………………….. g of OH^-

1000mL of water sample contains =(x)×10 =…………………….. g of OH^-

1000mL water sample contains = (x)× 10 × 1000 =…………………….. mg of OH^-

1000mL water sample contains =…………………….. mg of OH^-

(ii) Alkalinity due to if p<(l)/2M)

Step 1:

1000 mL of 1N H_2SO_4 = 30g CO_3^{2-}

[2P-M] mL of 0.01N H_2SO_4=$\frac{30\times0.01\times[2P-M]}{1000}$g of CO_3^{2-}

=$\frac{30\times0.01\times.....}{1000}$=............................... (y) g of CO_3^{2-}

Step 2:

100mL of water sample contains =(y) g = …………………….. g of CO_3^{2-}

1000mL of water sample contains =(y) ×10 =……………. …….. g of CO_3^{2-}

1000mL water sample contains =(y) ×10× 10000 = ……… …. mg of CO_3^{2-}

1000mL water sample contains = …………………….. mg of CO_3^{2-}

(iii) Alkalinity due to HC03$^-$ $(if\ p < \frac{1}{2M})$

Step 1 :

1000 mL of 1N H_2SO_4 =61g HC03-

[2P-M] mL of 0.01N H_2SO_4 =$\frac{61\times0.01\times[2P-M]}{1000}$ g of HCO_3^-

=$\frac{61\times0.01\times.....}{1000}$=…………..(z)g of HCO_3^-

Step 2 :

100 mL of water sample contains =(z) g =........................... g of HCO_3^-
1000 mL of water sample contains=(z)× 10 =........................... g of HCO_3^-
1000 mL water sample contains=(z) ×10× 1000 =........................... mg of HCO_3^-

1000 mL water sample contains =...................... mg of HCO_3^-

7.0 RESULTS

1. Alkalinity due to OH^- =...................... mg/L
2. Alkalinity due to $CO3^{2-}$ =...................... mg/L
3. Alkalinity due to $HCO3^-$ =...................... mg/L

8.0 CONCLUSION

....

PRACTICAL RELATED QUESTION

1. **What is alkalinity?**

2. **What are the ions responsible for alkalinity to a water?**

3. **What are the indicators used in the titration of alkalinity?**

4. **What is the colour change of phenolphthalein indicator in alkalinity titration?**

5. **What are the reactions gets completed during the phenolphthalein end point?**

6. **Why OH and HCO_3 ions cannot exist together in water?**

7. **What is the colour of methyl orange in a water sample having alkalinity?**

8. What are the ions gets neutralised in methyl orange end point and write the reaction?

9. How is alkalinity expressed?

10. What is the p^H range both in acidic and basic conditions of a solution?

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Roll no……………………

PERCENTAGE OF IRON IN HEMATITE

EXPERIMENT NO.06

1.0 AIM OF THE EXPERIMENT

Determine the percentage of iron in haematite ore by standard KMn04 solution.

2.0 HYH SET UP

2.1 LIST OF APPARATUS REQUIRED:-

1. Burette (50 ml)
2. pipette (10 ml)
3. round bottom flask (250 ml)
4. wire gauze, conical flask (100 ml)
5. Volumetric flask (250 ml)

2.2 LIST OF CHEMICALS REQUIRED:-

- $KMnO_4$
- dilute H_2SO_4
- zinc granules
- sample ore material
- oxalic acid
- HCI

3.0 THEORY

Haematite is an important ore of iron. For determination of iron in the given ore sample, the ore sample is dissolved in dilute HCI or dilute H2S04. Iron is converted to ferric or ferrous salt solution with liberation of hydrogen gas. Reducing agents used for converting Fe^3+ to Fe^2+ are metallic zinc or stannous chloride. The Fe^2+ present in the solution can be determined by titrating with standard KMn04 solution.

$$MnO_4^- + 8H^+ + Fe^{2+} \longrightarrow Mn^{2+} + Fe^{3+} + 4H_2O$$

4.0 PROCEDURE

PREPARATION OF SAMPLE SOLUTION

1. Weigh 1 g of ore and transfer it into 250 ml round bottom flask.
2. Add 100 ml of dilute H_2SO_4 into the round bottom flask and gently heat on wire gauze till the sample dissolve completely.
3. Add few granules of zinc when sample dissolves.
4. Cool it and transfer in to 250 ml volumetric flask, dilute the solution up to mark using distilled water.

STANDARDIZATION OF $KMNO_4$

1. Prepare 0.1 N $KMnO_4$ [refer preparation of normal solution.
2. Pipette out 10 ml of 0. 1N oxalic acid in conical flask.
3. Add 10 ml of dilute H_2SO_4 and heat the solution up to 70^0 C
4. Titrate the above solution against standard KMn04 solution till light pink colour appears
5. Repeat the procedure for three consecutive readings.

DETERMINATION OF PERCENTAGE OF IRON

1. Rinse and fill the burette with standard $KMnO_4$ solution.
2. Pipette out 10 ml of sample solution in conical flask. Add 10 ml of dilute H_2SO_4 solution
3. Titrate the above solution against standard $KMnO_4$ solution till light pink colour appears
4. Repeat the procedure for three consecutive reading.

5.0 OBSERVATIONS

Standardization of KMn04			Determination of % of Iron		
Sr.no	Observation	Reading	Sr.no	Observation	Reading
1	The solution in burette		1	The solution in burette	
2	Solution by pipette		2	Solution by pipette	
3	Indicator used		3	Indicator used	
4	Endpoint		4	Endpoint	

TABLE 1: STANDARDIZATION OF KMN04

Readings	Pilot Reading	1	2	3	V2=Mean Difference = (1 +2+3)/3
Initial Burette Reading (IBR)					
Final Burette Reading (FBR)					
Difference (FBR-IBR)					

TABLE 2: DETERMINATION OF PERCENTAGE OF IRON

Readings	Pilot Reading	1	2	3	V3=Mean Difference = (1 +2+3)/3
Initial Burette Reading					
Final Burette Reading					
Difference (FBR-IBR)					

6.0 CALCULATION

STANDARDIZATION OF $KMNO_4$

$N_1V_1=N_2V_2$

N_1 = Normality of oxalic acid 0. 1N

V_1 =Volume of oxalic acid 10mL

N_2 = Normality of $KMnO_4$

V_2 =Volume of KMn04 from table 1

$$N_2 = \frac{Normality\ of\ KMnO_4 \times Volume\ of\ oxalic\ acid}{Volume\ of\ KMnO_4\ from\ table\ 1}$$

$$= \frac{0.1\times10}{Concordant\ burette\ reading\ from\ table\ 1} = \frac{1}{V_2} = \text{.................} = \text{.................}$$

DETERMINATION OF PERCENTAGE OF IRON

step 1 1000mL 1N $KMnO_4$= 56g of Fe

$$V_3\ \text{mL}\ N_2\ \text{N}\ KMnO_4 \equiv \frac{(56 \times V_3 \times N_2)}{1000}\ \text{of Fe} = \frac{(56\ \text{x.}\ ......}{1000} = \text{...........(y) g of Fe}$$

Step 2 10 mL of ore sample ≡ y g of Fe

$$250\ \text{mL of ore sample} \equiv \frac{(y\times250)}{10} = y \times 25\ g\ of\ Fe = \text{.........}\times 25 = \text{.........}\ \text{(z)g Fe}$$

Step 3 1 g of Sample (z)g Fe

$$100\ \text{g of sample} \equiv \frac{(z\times1000)}{1} = \text{...........g of Fe}$$

7.0 RESULT

Percentage of iron in haematite ore sample =………...%

CONCLUSIONS

………

PRACTICAL RELATED QUESTION

1. Name the ores of ion?
2. What is haematite, and why is it significant in iron estimation?
3. What is the purpose of adding HCl to the hematite ore sample before titration?
4. Why is KMnO4 solution used as a titrant in this experiment?
5. What is the end point of the titration?
6. The end point is reached when the pink colour of the KMnO4 solution persists after adding a drop of the titrant.
7. How do you ensure that the KMnO4 solution is standardized?
8. Why is it important to use a burette to add the KMnO4 solution?
9. What is the purpose of adding a few drops of H2SO4 to the titration flask?
10. How do you handle the ore sample to prevent contamination?
11. What is the significance of repeating the titration?

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IODOMETRIC ESTIMATION OF COPPER

EXPERIMENT NO.07

1.0 AIM OF THE EXPERIMENT

Estimate Iodometrically copper in the copper pyrite ore.

2.0 EXPERIMENTAL SET UP

2.1 LIST OF APPARATUS REQUIRED:-

1. Standard flask
2. Funnel
3. wash bottle
4. Beaker
5. Dropper
6. Spatula
7. standard flask

2.2 LIST OF CHEMICALS REQUIRED:-

- Potassium dichromate ($K_2Cr_2O_7$)
- sodium thiosulphate ($Na_2S_2O_3$)

3.0 THEORY

Due to ductility, electrical and thermal conductivity, copper is used in production of electrical conductors, switches, transformers and telecommunications. Hence its extraction and percentage calculations plays a vital role. Copper can be estimated through iodometric titration in which oxidizing agents liberates iodine which is titrated with standard solution of reducing agent called as indirect or back titration. In this titration iodine is liberated by oxidizing agent Cu^2+, which is titrated against reducing agent sodium thiosulphate solution using starch as an indicator. The reaction between potassium iodide solution and potassium dichromate solution in acidic medium liberates iodine. Sodium thiosulphate solution (hypo solution) reduces liberated iodine to iodide.

CHEMICAL REACTIONS:

$K_2Cr_2O_7 + 6KI + 14\ HCl \rightarrow 2CrCl_3 + 8KCI + 3I_2 + 7H_2O$

Orange Green

$Na_2S_2O_3 + 3I_2 \rightarrow 3Na_2S_4O_6 + 6NaI$

$K_2Cr_2O_7 = 6Na_2S_2O_3$

Oxidising agent $K_2Cr_2O_7$

Reducing agent $Na_2S_2O_3, 5H_2O$

$CuSO_4 + 4KI \rightarrow Cu_2I_2 + 2K_4SO_4 + I_2$

Blue White ppt

$Na_2S_2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2NaI$

$2CuSO_4 = 2Na_2S_2O_3$

Oxidising agent Cu^{2}+

Reducing agent $Na_2S_2O_3, 5H_2O$

4.0PROCEDURE-1

PREPARATION OF STANDARD COPPER SAMPLE (BRASS SOLUTION / TEST SOLUTION)

1. Clean the glasswares thoroughly before use.
2. Weigh 1 g of Copper sample, add to it 1:1 test solution of HNO_3 boil the mixture till brass dissolve completely.
3. Add 1 g of urea to expel the dissolved NO_2 completely. Boil till white vapors appears.
4. Add ammonia solution till deep blue colour appears.
5. Add acetic acid till light blue colour appears. It is added to neutralize acid. For liberation of iodine, slight acidic environment is required.
6. Transfer the reaction mixture in 250m1 standard flask and dilute up to the mark.

PREPARATION OF 250ML, 0.1N(O.1/6M) POTASSIUM DICHROMATE SOLUTION

Volume of Potassium dichromate solution (v) = 250mL

Strength of potassium dichromate solution M= 0.1/6 M

Molar Mass of potassium dichromate ($K_2Cr_2O_7$) =294 g mol-I

Weigh of $K_2Cr_20_7$ for the preparation of 250mL 0.1 / 6 M solution

$$= \frac{Molarity \times Volume \times Molar\ mass}{6 \times 1000} = 1.225\text{g}$$

1. Weigh out accurately 1.225g of potassium dichromate using electronic balance.
2. Transfer solid potassium dichromate in standard volumetric flask
3. Add about 100mL of distilled water and dissolve solid potassium dichromate.
4. Dilute the solution up to the mark i.e. to make it 250 ml.

PREPARATION OF 0.1 N SODIUM THIOSULPHATE SOLUTION

1. Weigh out accurately 6.2 g of sodium thiosulphate
2. Transfer sodium thiosulphate in another 250 ml standard flask
3. Add about 100 ml of distilled water and dissolve solid sodium thiosulphate.
4. Dilute the solution upto the mark i.e. to make it 250m1.

PROCEDURE -2

STEP 1: STANDARDIZATION OF POTASSIUM DICHROMATE SOLUTION

1. Pipette out 20 ml of potassium dichromate solution into the conical flask.
2. Add 10 ml of 20% KI solution in the conical flask which contains potassium dichromate solution.
3. Add 5 ml of conc. HCI to same flask
4. Close the conical flask with watch glass and put the conical flask in dark place for 5 min.
5. Rinse and clean the burette with sodium thiosulphate solution (hypo solution)
6. Place conical flask in the dark and add 150 ml of distilled water.
7. Titrate this solution against sodium thiosulphate till it becomes pale yellow.
8. Add 2 ml of starch as an indicator.
9. Continue the titration till the solution becomes clear green.
10. Note down the reading (end point of titration)

STEP 2: DETERMINATION OF PERCENTAGE OF COPPER

1. Pipette out 20 ml of copper solution.
2. Add few drops of ammonia solution in copper solution till it becomes turbid (forms ppt)
3. Add few drops of acetic acid in copper solution till it dissolves all precipitate.
4. Add 10mL of 20% KI solution
5. Cover the conical flask with watch glass and put the conical flask in dark place for 5 min.
6. Add 100 ml of distilled water

7. Titrate this solution against sodium thiosulphate till it becomes pale yellow.
8. Add 2 ml of starch to pale yellow solution (colour changes from pale yellow to dark blue/ violet ppt)
9. Continue the titration with sodium thiosulphate till colour changes from dark blue to milky white solution
10. Add 20 ml 2% of sodium thiocyanate solution
11. Titrate this till it becomes white ppt.
12. Note the end point of titration.

5.0 OBSERVATIONS AND CALCULATIONS

OBSERVATION TABLE 1: STANDARDIZATION OF POTASSIUM DICHROMATE SOLUTION

Readings Pilot Reading	1	2	3	Vx=Mean Difference =(1+2+3)/3
Initial Burette Reading (IBR)				
Final Burette Reading (FBR)				
Difference (FBR-IBR)				

$1K_2Cr_2O_7 \equiv 6Na_2S_2O_3$

M_1=0.1M, V_1=20mL, n_1=1; M_2 =? V_2=V_x, n_2=6

$\frac{M_1V_1}{n_1} = \frac{M_2V_2}{n_2}=$

$$M_2 = \frac{M_1 \times V_1 \times n_2}{n_1 \times n_2} = \frac{0.1 \times 20 \times 6}{6 \times Burette\ reading\ V_x} = \frac{2}{6 \times Burette\ reading\ Vx} = \frac{\dots\dots\dots\dots\dots\dots\dots}{\dots\dots\dots\dots\dots\dots\dots}$$

Molarity of hypo solution = (Mhypo) =……..M

OBSERVATION TABLE 2: DETERMINATION OF PERCENTAGE OF COPPER

Readings Pilot Reading	1	2	3	Vy=Mean Difference =(1+2+3)/3
Initial Burette Reading (IBR)				
Final Burette Reading (FBR)				
Difference (FBR-IBR)				

$2Na_2S_2O_3 = 2CuSO_4$

$M_2 =$ Mhypo, $V_2 =$ Constant burette reading (Vy), $n_2=2$; $M_3=?$ $V_3=20$mL, $n_3=2$

$$\frac{M_2V_2}{n_2} = \frac{M_3V_3}{n_2}$$

$$\frac{M_2 \times V_2 \times n_2}{n_2 \times n_3} = \frac{Mhypo \times burette\ reading\ (Vy) \times n_3}{n_2 \times V_3}$$

$$= \frac{Mhypo \times burette\ reading\ (Vy) \times 2}{2 \times 20} = \frac{\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots}{\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots}$$

Molarity of copper $Cu^{2}+$ solution = M_3 =……….M

Amount of Cu^{2+} in 100 mL solution $= \frac{M_3 \times Atomic\ weight\ of\ copper \times 100}{1000}$

$= \frac{M_3 \times 63.5 \times 100}{1000} =$……………………..

Amount of Cu^{2+} in 100 mL solution $= z = M_3 \times 6.35$

1 g sample = z g Cu

100 g sample = z x 100 =…………..x 100 =……….

6.0 RESULTS

Percentage of copper present in given sample is =……….%

7.0 CONCLUSION

……………………………………………………………………………

PRACTICAL RELATED QUESTION

1. What is iodometric titration?

2. Name some oxidising agents.

3. Why hypo is commonly used as a reducing agent?

4. Which indicator is used in iodometry?

5. Why is blue colour formed in the iodometric titration?

6. Why is blue colour disappeared in the titration?

7. What is the equivalent weight of copper?

8. Why a burette with rubber tap should not be used in iodimetry?

9. Why iodine solution is stored in coloured bottles?

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Roll no……………………

ACID-BASE TITRATION

EXPERIMENT NO.08

1.0 AIM OF THE EXPERIMENT

To Determine total acid number (TAN) of given oil.

2.0 EXPERIMENTAL SET UP

2.1 LIST OF APPARATUS REQUIRED:-

1. Conical flask (100 ml)
2. Burette (50 ml)
3. Pipette (10 ml)
4. Weighing balance (0.0001 mg sensitivity)

2.2 LIST OF CHEMICALS REQUIRED:-

- Oil sample
- phenolphthalein indicator
- KOH

3.0 PROCEDURE

1. Weigh l g of oil sample in conical flask.
2. Add 50ml of ethyl alcohol to oil to shake the solution to dissolve oil completely.
3. Add 2-3 drops of phenolphthalein indicator into the solution in conical flask.
4. Rinse and fill the burette with 0.01 N KOH solution.
5. Titrate the solution in conical flask with 0.01 N KOH solution till colour changes from colourless to permanent light pink.
6. Repeat the procedure for three consecutive burette readings.

4.0 OBSERVATION

1. Solution in burette…………………………………….
2. Solution in conical flask…………………………………
3. Indicator……………………………………………………
4. End Point…………………………………………………

5.0 OBSERVATION TABLE

Readings	Pilot Reading	1	2	3	Mean Difference =(1+2+3)/3
Initial Burette Reading (IBR)					
Final Burette Reading (FBR)					
Difference (FBR-IBR)					

1000 ml of 1N KOH = 56.119 g of KOH

1 ml of 1N KOH = 5 6.119 mg of KOH

1 ml of 0.01 N KOH = 56.119 x 0.01 mg of KOH

(mean difference) ml of 0.01 N KOH = 56.119 x 0.01 x (mean difference ml) mg of KOH

Acid value of oil sample= 56.119×0.01 ×(mean difference ml) milligram of KOH / 1g of Oil

=...

Acid Value = ___________.

6.0 RESULT

Acid Value of 1g of given sample of oil is................................

7.0 CONCLUSION

....

PRACTICAL RELATED QUESTION

1. **What is the Total Acid Number (TAN) and why is it important in the context of oil analysis?**

2. **How is the Total Acid Number (TAN) typically determined in the laboratory?**

3. **What is the Total Acid Number (TAN) of oil, and how does it affect the performance and maintenance of machinery?**

4. **List available lubricant oil in market.**

5. **Give significance of acid value of an oil.**

6. **Relate the TAN with degradation of lubricant.**

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Roll no……………………

CONDUCTIVITY OF WATER

EXPERIMENT NO.09

1.0 AIM OF THE EXPERIMENT

To Determine the conductivity of given water sample

2.0 EXPERIMENTAL SET UP

2.1 LIST OF APPARATUS REQUIRED

1. Conductivity meter (Range-O to 20, 200gS accuracy ± 1% range
2. Magnetic stirrer (250-1250 rpm)
3. Beaker (100ml)
4. Glass rod
5. Standard flask

2.2 LIST OF CHEMICALS REQUIRED

- 0.1 N KCI solution
- water sample

3.0 THEORY

Conductivity is the ability of a solution to conduct electric current. Conductivity is measured with a probe and meter. Conductivity (G) is inverse of resistivity (R) is determined from the voltage and current values according to ohms law R=V/I then I/R=I/V. The meter converts the probe measurement to micro mhos per centimeter and displays the result.

Conductivity is measured in micromho/cm(μmho/cm) or millimho/cm (mmho/cm) or milisiemen/m (mS/m) 1mS/m=10 μmho/cm

4.0 RACTICAL SETUP

(Drawing/Sketch/Circuit Diagram/Work Situation)

5.0 PROCEDURE

1. Measure 50 mL of distilled water and transfer it to the beaker.
2. Weigh 0.7456 g of Potassium chloride.
3. Transfer the 0.7456 g of potassium chloride to the beaker containing distilled water. Stir continuously by the glass rod until it dissolves thoroughly.
4. Transfer the contents to the 100 mL standard flask.
5. Make up the volume up to 100 mL. By adding distilled water and shake the contents well. The prepared solution is used to calibrate the conductivity meter.
6. Take 0. IN Potassium Chloride in a beaker. Switch on the magnetic stirrer and place the beaker on the stirrer. Insert the magnetic bead in the beaker.
7. Place the electrode inside the solution. Select the calibration button and by using up and down keys, adjust the conductivity of the 0.1 N potassium chloride solution to 14.12 millisiemens /cm at 30^0 C.
8. Now the conductivity meter is ready for the measurement of samples.

PROCEDURE FOR TESTING OF WATER SAMPLE

1. Rinse the electrode thoroughly with deionised water and carefully wipe with a tissue paper.
2. Measure 200 mL of water sample and transfer it to a beaker and place it on the magnetic stirrer.
3. Dip the electrode into the sample solution taken in a beaker and wait for a steady reading.
4. Make sure that the instrument is giving stable reading.
5. Note down the reading in the display directly, which is expressed in millisiemens.

6.0OBSERVATION AND CALCULATION

Conductivity of given sample is…………….............millisiemens.

7.0 RESULT

Conductivity of given sample is…………….............millisiemens.

8.0 CONCLUSION

……………………………………………………………………………………

PRACTICAL RELATED QUESTION

1. **What is electrical conductivity, and how is it related to water?**
2. **What is the normal conductivity of water?**
3. **How does the presence of dissolved salts affect the conductivity of water?**
4. **Can you explain the difference between distilled water and tap water in terms of conductivity?**
5. **What factors can influence the conductivity of natural water bodies like rivers or lakes?**
6. **Why is it important to monitor the conductivity of water in environmental studies?**
7. **What is the role of ion concentration in determining water conductivity?**
8. **Can you describe how you would measure the conductivity of water in a lab setting?**
9. **What are some practical applications of measuring water conductivity?**

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Roll no……………………

MEASUREMENT OF EMF USING DANIEL CELL

EXPERIMENT NO.10

1.0 AIM OF THE EXPERIMENT

To Determine the emf using an electrochemical cell (Daniel Cell).

2.0 EXPERIMENTAL SET UP

2.1 LIST OF APPARATUS REQUIRED:-

1. Multimeter
2. voltmeter
3. connecting wires
4. Beaker (1000mL)

2.2 LIST OF CHEMICALS REQUIRED:-

1. Copper sulfate ($CuSO_4$)
2. Zinc Sulfate ($ZnSO_4$)
3. Zn Rod
4. Cu Rod
5. Salt bridge

3.0 THEORY

The potential difference between the two electrodes in a galvanic cell is called a cell potential or emf of the cell. It is measured in volts. Daniel cell is also known as a galvanic cell or voltaic cell. This cell is an example of an electrochemical cell. Generally, rods or plates are termed as electrodes. Each electrode is immersed in its metal salt solution, and these forms half-cell. Two half-cells are separated by a salt bridge or porous partition. The solution which conducts electricity either in a molten state or in an aqueous solution is called electrolytes. One electrode acts as an anode where oxidation takes place. When two metals are connected, the metal with higher activity acts as an anode while the other metal acts as the cathode.

Electrode reactions in daniel cell

Reaction at anode $Zn \longrightarrow Zn^{2+} + 2e^-$ Oxidation Reaction

Reaction at cathode $Cu^{2+} + 2e^- \longrightarrow Cu$ Reduction Reaction

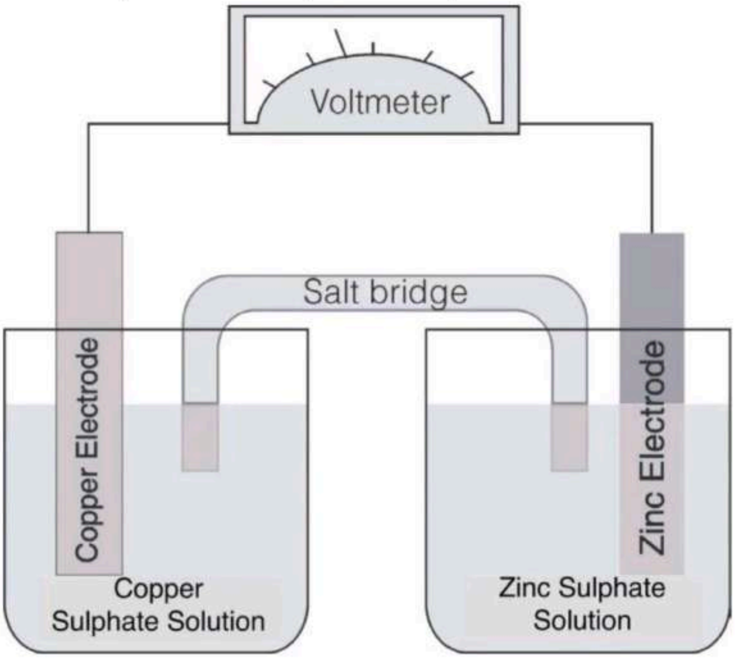
$Zn + Cu^{2+} \longrightarrow Zn^{2+} + Cu$ Redox Reaction

Daniel cell is represented as Zn(s) I $ZnSO_4$ (aq)I I $CuSO_4$ (aq) I Cu(s)

Voltage produced at daniel cell is the sum of reduction and oxidation voltage developed across the electrode is written as E^O Cell = E^O Reduction + E^O Oxidation

4.0 PRACTICAL SETUP

(DRAWING/SKETCH/CIRCUIT DIAGRAM/WORK SITUATION)



A primary cell with a constant electromotive force of about 1.1 volts having as its electrodes copper in a copper sulfate solution and zinc in dilute sulfuric acid or zinc sulfate, the two solutions being separated by a porous partition. In Daniel cell Cu and Zn electrodes are immersed in $CuSO_4$ and $ZnSO_4$ solution respectively. Zn acts as anode and Cu acts as cathode. Oxidation takes place at Zn and reduction takes place at the cathode. The Zn ions passed in solution. As two electrodes are connected using an external wire, the electrons produced by oxidation of Zn move through the wire and are collected at the copper electrode. Cupric ions present in the solution (electrolyte) accept electrons and are discharged at the cathode.

5.0 PROCEDURE

1. Place Zn rod in $ZnSO_4$ solution and Cu in $CuSO_4$ solution.
2. Connect Zn rod to one end and Cu rod to another end of the multimeter.
3. Place salt bridge in both the solution. A Salt bridge is an inverted 'U' tube filled with the saturated solution of an inert electrolyte KCl/KN03 containing agar-agar, it releases ions into the electrolyte so that the whole solution becomes electrically neutral.
4. Take the concentration of ZnS04 and CuS04 solution as shown in observation table.
5. Prepare five different concentrations of ZnS04 and CuS04, respectively.
6. Connect two beakers with combinations shown in the observation table.
7. Record the emf developed across these two half cells.

6.0 OBSERVATION

Cell No.	Concentration of ZnS04	Concentration of CuS04	EMF produced (in Volts)
1	2Molar (M)	2M	
2	1M	2M	
3	0.5M	2M	
4	0.25M	2M	
5	0.0125M	2M	
6	2M	1M	
7	2M	0.5M	
8	2M	0.25M	
9	2M	0.0125M	
10	1M	1M	

7.0 RESULT

1. Emf developed in cell 1 is =………………….. . Volts
2. Emf developed in cell 2 is =………………… . Volts
3. Emf developed in cell 3 is =………………… . Volts
4. Emf developed in cell 4 is =………………… . Volts
5. Emf developed in cell 5 is =………………….. . Volts
6. Emf developed in cell 6 is =………………… . Volts
7. Emf developed in cell 7 is =………………… . Volts
8. Emf developed in cell 8 is =………………… . Volts
9. Emf developed in cell 9 is = ..………………......Volts
10. Emf developed in cell 10 is =…………………Volts

8.0 CONCLUSION

…………………………………………………………………………………………………

PRACTICAL RELATED QUESTION

1. What is oxidation?

2. What is reduction?

3. What is a voltaic cell or electrochemical cell?

4. Define the term emf?

5. What is a half cell ?

6. Mention the names of anode and cathode of a Daniell cell ?

7. Can copper be oxidised by zinc ions ?

8. What is a salt bridge ?

9. Name the electrolytes that can be used in salt bridge.

10. What is the direction of flow of electrons in an electrochemical cell ?

11. What is the direction of flow of current in an electrochemical cell ?

12. What is the effect of [Zn^{2+}] on emf of the cell Zn | Zn^{2+}|| Cu^{2+} | Cu ?

13. What is the effect of [Cu^{2+}] on emf of the cell Zn | Zn^{2+}|| Cu^{2+} | Cu ?

14. What factor is kept in mind while selecting an electrolytic solution for the construction of a salt bridge ?

15. Is it possible to measure the single electrode potential ?

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