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LIST OF EXPERIMENTS

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1. Estimation of Hardness of water by Complexometry using EDTA .
2. Estimation of Fe^{+2} by Dichrometry.
3. Estimation of Ferrous by Permanganometry.

II. Conductometry:

1. Estimation of the concentration of an acid by Conductometry.

III. Potentiometry:

1. Estimation of the amount of Fe^{+2} by Potentiomentry.
2. Estimation of the concentration of an acid by Potentiomentry.

IV.Preparations:

1. Preparation of Bakelite.
2. Preparation Nylon – 6.

V. Lubricants:

1. Estimation of acid value of given lubricant oil.
2. Estimation of Viscosity of lubricant oil using Ostwald's Viscometer.

VI. Virtual lab experiments

1. Construction of Fuel cell and its working.
2. Smart materials for Biomedical applications
3. Batteries for electrical vehicles.
4. Functioning of solar cell and its applications.

GLOSSARY:

TITRATION :

The process of determining the strength of an unknown solution (taken in the conical flask) by allowing it to react with a standard solution (solution of known strength taken in the burette) is known as titration. The completion of the reaction is monitored by the use of an indicator.

TITRANT:

The solution containing a known weight (know concentration) of a substance is called a titrant.

TITRATE:

The solution which contains a substance to be estimated (unknown concentration) is known as Titrate.

STANDARD SOLUTION :

The exact amount of the substance present in a definite volume of the solution is known as standard solution.

Concentration of a solution is expressed in various ways :

1. NORMALITY (N) :

The normality of a solution is the number of gram equivalent of the solute per litre of the solution.

Normality (N) = No. of gram equivalent of solute / volume of solution in 1000 ml

2. MOLARITY (M) :

The molarity of a solution is the number of moles of the solute per litre of the solution

Molarity (M) = No. of moles of solute / volume of solution in 1000ml

3. MOLALITY (m) :

The molality of a solution is the number of moles of the solute per 1000g of the solvent

No. of moles of solute Molality (m) = Mass of solvent in Kg

INDICATOR:

It is substance which when added to the titrate, indicator the completion of reaction by changing its color at the end point.

END POINT:

This is the point in the titration, which indicates the completion of a chemical reaction.

STANDARDIZATION :

It means determination of the strength of an unknown solution with the help of a standard solution.

ESTIMATION :

To find out the amount of a substance present in certain a volume of the given solution.

SOLUTE:

The substance, which dissolves into the solution, is called as a solute.

NORMAL SOLUTION (1N SOLUTION):

A normal solution is a solution containing one gram equivalent weight of the solute in one litre of the solvent.8

DECI NORMAL SOLUTION (N/10 SOLUTION) :

A solution containing one tenth of a (1\10) gram equivalent of the solute in one litre of the solution is known as deci-normal solution.

PRINCIPLE INVOLVED IN VOLUMETRIC ANALYSIS :

Volumetric analysis is based on the law of volumetric analysis. When two solutions completely react with each other, the product of volume and normality of one solution will be equal to the product of volume and normality of the other solution.

$$V_1N_1 = V_2N_2$$

V_1=Volume of the first solution

N_1=Normality of the first solution

V_2=Volume of the second solution

N_2=Normality of the second solution

IMPORTANT CALCULATIONS:

1) Normality (strength) of a solution = (Weight of the solute in one liter) / (Equivalent weight of the solute)
2) Weight of the solute in one liter = (Equivalent weight of the solute) x (Normality of the solution)
3) Weight of solute present in y ml of the solution = Equivalent weight of solute Normality of the solution x (y/1000)

ABBREVATIONS USED IN VOLUMETRIC ANALYSIS:

1. V = Volume
2. ml = Millilitre
3. gm = grams
4. N = Normality
5. Conc = Concentration
6. di l=diluted
7. Ppm =Parts per million
8. M = Molarity
9. m = molality

P R E C A U T I O N S

1. Wear apron before entering lab.
2. Do not push or shove other students in the lab or classroom.
3. Do not crowd lab stools.
4. Be careful with Bunsen burners, Hot Plates, and other heat sources.
5. Keep all papers away from heat sources.
6. Clean lab or classroom area after experiments (glassware, stools, lab bench and sink)
7. Return all borrowed materials to their appropriate storage place as directed by the teacher.
8. Never mix chemicals without permission or instruction.
9. Do not taste or ingest any chemical or specimen in the laboratory.
10. Never smell directly from any container – always waft contents.
11. Never look directly into a test tube –always view test tube from the side.
12. When heating a container always point it away from self and others.
13. Place broken glass or other lab materials in the container designated by the teacher.
14. Never add water to an acid-always add acids to water.
15. Be careful when using electricity.
16. If you spill a small amount of acids, bases, or other caustic materials on your skin wash immediately with lots of water at the nearest sink.
17. Glass waste must be put in the glass waste container.
18. Smoking, eating, chewing gum or tobacco, drinking, or applying cosmetics are not allowed in the lab.
19. Clamp all apparatus firmly, especially when heating, cooling.

20. Do not put anything in your mouth while working in the chemistry lab.
21. Wash hands frequently while in the lab and at the end of each lab period.

EXPERIMENT -1

ESTIMATION OF HARDNESS OF WATER BY EDTA METHOD

AIM : To estimate the amount of temporary and permanent hardness of water by the EDTA method.

APPARATUS: Burette, volumetric flask, conical flask, pipette, dropper.

CHEMICALS: $MgSO_4$,EDTA, Eriochrome black-T indicator (solochrome), P^H10 Buffer solution, water sample.

PRINCIPLE : Temporary hardness of water is due to the dissolved salts of calcium andmagnesium bicarbonates. On heating these salts decompose to give insoluble carbonates and hydroxides.

$$Ca(HCO_3)_2 \xrightarrow{\Delta} CaCo_3\downarrow + H_2O + CO_2\uparrow$$

$$Mg(HCO_3)_2 \xrightarrow{\Delta} Mg(OH)_2\downarrow + 2CO_2\uparrow$$

Thus temporary hardness can be removed by boiling the sample of hard water.

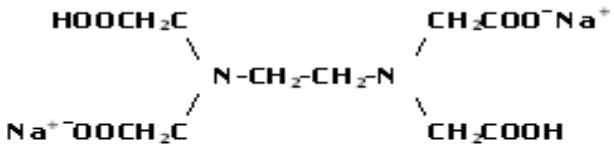
When EBT is added to hard water buffered to a P^H of about 10, a wine red unstable complex is formed. Thus

Mg+2 + EBT ⟶ (Mg-EBT) complex
(wine red)

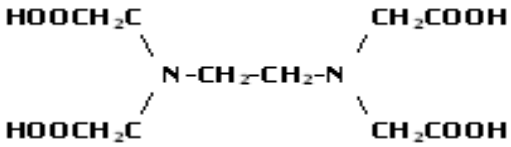
During the course of titration against EDTA solution, EDTA combines with Mg+2 ions form stable complex and releasing EBT.However, when nearly all Mg+2 ions have formed complex, then next drop of EDTA added displaces the EBT indicator from complex and wine red colour changes to blue colour. Thus, at end point, (Colourless)

(Mg – EBT) complex+EDTA ⟶ (Mg-EDTA) complex + EBT
(wine red) (Colourless) (blue)

DISODIUM SALT OF EDTA



STRUCTURE OF EDTA



PROCEDURE:

1. Preparation of standard MgSO4solution:

Weigh accurately about 0.25g of pure crystalline MgSO4.7H2O in to a clear 100ml standard flask, which has been cleaned with water and rinsed

with distilled water. Dissolve it in minimum amount of distilled water and make up to mark by adding more distilled water. Shake the solution well for uniform concentration. Calculate its molarity.

$$\text{Molarity of } MgSO_4 = \frac{0.25 \times 1000}{246.5 \times 100} = 0.01M$$

Mol. Wt of the salt = 246.5g

2. Standardization of EDTA:

Rinse a clean burette, first with distilled water and then with the EDTA solution and fill it with the same solution up to a convenient mark. Rinse a clean pipette with distilled water and then with the prepared MgSO4 solution. Pipette out 10ml of MgSO4 solution into a clean conical flask which has been cleaned with water and rinsed with distilled water. Add 2ml of the buffer solution, 3-4 drops of Erio-chrome black-T indicator. Titrate this solution with EDTA solution taken in the burette till the colour changes from wine red to blue. Note down reading. Repeat the titration for concurrent readings. Calculate the molarity of EDTA.

S. No.	Volume of $MgSO_4$ solution	Burette Reading		Volume of EDTA solution
		Initial	Final	
1	10 ml	0	10	10
2	10 ml	10	20	10
3	10 ml	20	30	10

Volume of $MgSO_4$ solution (V_1) = 10ml
Volume of EDTA solution (V_2) = 10
Molarity of MgSO4 solution (M_1) = 0.01M
Molarity of EDTA solution (M_2) = ?

$$M_1V_1 = M_2V_2$$

$$M_2 = \frac{0.01 \times 10}{10}$$

$$= \quad M$$

1. **Estimation of total hardness of water:**

Pipette out 20 ml of the sample of hard water into a clean conical flask, which ha been rinsed with distilled water. Add 2ml of P**H** 10 buffer solution, and 3 drops of Eriochrome black-T indicator. Titrate this solution with the standardized EDTA solution until the colour changes from wine red to blue. Note down the reading, repeat the titration for concurrent titre value and calculate the molarity and then the amount of hardness of the sample of hard water.

S. No.	Volume of water	Burette Reading		Volume of EDTA
		Initial	Final	

				solution
1	20 ml	0	6	6
2	20 ml	6	12	6
3	20 ml	12	18	6

Volume of tap water solution (V_3) = 10ml
Volume of EDTA solution (V_4) = 6
Molarity of tap water solution (M_3) = ?
Molarity of EDTA solution (M_4) = 0.01M

$$M_3V_3 = M_4V_4$$

$$M_3 = \frac{0.01 \times 6}{10}$$

$$= 0.006 \text{ M}$$

Total hardness of water = molarity × 10^5

= PPM

2.Estimation of permanent hardness of water :

Place 250ml of the sample of water in a 500ml beaker and boil gently for 20-30 minutes. Cool and filter, collect the filtrate into a 250ml standard flask. Pipette out 20ml of this solution into a clean conical flask and Add 2ml of P^H=10 buffer solution and 3 drops of Erio chrome black-T indicator. Titrate with the standardized EDTA solution until the colour changes from wine red to blue. Note down the reading, repeat the process to get at least two equal titre values. Calculate the permanent and then temporary hardness as parts per million of $CaCO_3$.

S. No.	**Volume of water**	**Burette Reading**		**Volume of EDTA solution**
		Initial	**Final**	
1	20 ml	0	3	3
2	20 ml	3	6	3
3	20 ml	6	9	3

Volume of boiled water solution (V_5) = 10ml
Volume of EDTA solution (V_6) = 6
Molarity of boiled water solution (M_5) = ?
Molarity of EDTA solution (M_6) = 0.01M

$$M_5V_5 = M_6V_6$$

$$M_5 = \frac{0.01 \times 3}{10}$$

= M

Permanent Hardness of water = molarity × 10^5

= PPM

3.Estimation of Temporary hardness of water :

Temporary hardness = Total hardness – Permanent hardness

RESULT:

1. The total hardness of given water sample is = PPM
2. The permanent hardness of given water sample is = PPM
3. The temporary hardness of given water sample is = PPM

VIVA QUESTIONS:

1.Define hardness of water?
2.What is the difference between hard water and soft water?
3.What are the salts responsible for the temporary and permanent hardness of water?
4.What are complex metric titrations?
5.What is meant by softening of water?
6.How the hardness of water is expressed?
7.Why do we express hardness of water in terms of calcium carbonate equivalent?
8.What is degree of hardness of water?
9.Why P^H=10 buffer solution is added during the determination of hardness of water?

Experiment-2

ESTIMATION OF FERROUS ION BY DICHROMETRY

Aim : Estimate the amount of ferrous iron in the whole of the given ferrous solution using external indicator (Diphenyl amine).
Apparatus: Beaker, pipette, 250 ml volumetric flask, Burette ,dropper.

Chemicals required: Potassium di chromate ($K_2Cr_2O_7$),Sulphuric acid (H_2SO_4), Diphenylamine, Ammonium Ferrous Sulphate $(NH_4)_2Fe(SO_4)_2.6H_2O$.

Principle : The potassium dichromate is an oxidizing agent and the reaction between potassium dichromate and ammonium ferrous sulphate is given below .

$6(NH_4)_2Fe(SO_4)_2+K_2Cr_2O_7 + 7H_2SO_4 \rightarrow 6(NH_4)_2SO_4+K_2SO_4+Cr_2(SO_4)_3 + 3Fe(SO_4)_3 + 7H_2O$

Equivalent mass of ferrous ion =55.85g

Procedure:

a)Preparation of Standard Potassium dichromate:

Weighed about 0.65g of $K_2Cr_2O_7$ accurately transferred into a 250ml standard flask. It is dissolved in water and made up to the mark.

b)Estimation of Ferrous iron :

The given solution is made up to 100ml. 20 ml of the made up solution is pipette out in to a clean conical flask and 20 ml dil sulphuric acid is added .Then one or two drops diphenylamine is added. The above colourless solution is titrated against standard potassium dichromate solution taken in the burette. The end point is the appearance of a blue violet colour. Repeat the titration to get concurrent values and the values are tabulated.

Observation and calculations:

S.NO	Volume of ammonium Ferrous sulphate solution (ml)	Burette reading (ml)		Volumeof $K_2Cr_2O_7$ (ml)
		Initial	Final	
1	20ml	0 ml	7	7
2	20ml	7	14	7
3	20ml	14	21	7

CALCULATION :

Volume of Mohrs salt (V1) = 20 ml

Normality of Mohrs salt (N1) = ?

Volume of Potassium dichromate (V2) = 7

Normality of potassium dichromate (N2) = 0.01N

$$N_1 = \frac{N2 \times V_2}{V_1}$$

$$N1=\frac{0.01\times7}{20}$$

$$N1=0.0035N$$

The amount of Fe^{2+} ion present in the given solution = N1 X 55.85

R E S U L T :

1. The normality of the given solution = ---------N
2. The amount of Fe^{2+}ion present in the whole of the given solution =---g/l

Viva questions:

1. How to prepare 500ml of 0.1N potassium dichromate?
2. Define redox reaction?
3. Write the formula of ammonium ferrous sulphate?
4. What is the name of the indicator in this Estimation ?
5. What is the structure of potassium dichromatre ?
6. What is the oxidation state of Cr in reactant and product sides?
7. What is the colour changes of this experiment?

EXPERIMENT-3

ESTIMATION OF FERROUS ION BY PERMANGANOMETRY

AIM: To Prepare the standard solution of oxalic acid and estimation of iron by potassium permanganate as an intermediate.

CHEMICALS: oxalic acid, potassium permanganate, concentrated sulphuric acid

Principle :

The potassium permanganate is an oxidizing agent and the reaction between potassium permanganate and ammonium ferrous sulphate is given below .

$10(NH_4)_2\,Fe(SO_4)_2 + 2KMnO_4 + 8H_2SO_4 \rightarrow 10(NH_4)_2SO_4 + K_2SO_4 + 2MnSO_4 + 5Fe_2(SO_4)_3 + 8H_2O$

Equivalent mass of ferrous ion =55.85g

PROCEDURE:

PREPARATION OF STANDARD OXALIC ACID SOLUTION:

Weigh out accurately 0.315gms of oxalic acid crystals in to a 100 ml standard flask dissolve the sample in little distilled water and make up the solution to the mark distilled water and shake the flask well for uniform concentration.

PREPARATION OF POTASSIUM PERMANGANATE SOLUTION:

Dissolve 1.58gms potassium permanganate in 1000 ml of distilled water and shake the flask well for uniform concentration.

PREPARATION OF 2 N SULPHURIC ACID SOLUTION:

56 ml concentrated sulphuric acid is added drop by drop slowly to 1000 ml of water taken in a beaker ,kept in a trough of water .Not directly added to water.

STANDARDISATION OF POTASSIUM PERMANGANATE SOLUTION:

Pipette out 20 ml of the oxalic acid solution in to a 250 ml conical flask and add 20 ml of 2N sulphuric acid. Heat the solution till fumes come out of the solution and titrate the hot solution with $KMnO_4$ taken in a burette. Pale pink colour is the end point. Repeat the titration to get concurrent values.Let the titre value be X ml.

S.NO	Volume of Oxalic acid solution (ml)	Burette reading		Volume of $KMnO_4$ (ml)
		Initial	Final	
1	20	0	20	20
2	20	20	40	20
3	20	40	60	20

Calculations:

Titration -I

Normality of oxalic acid (N_1) = 0.1N

Normality of $KMnO_4$ (N_2) =?

Volume of oxalic acid (V_1) = 20ml

Volume of $KMnO_4$ (V_2)= X

$$N_1V_1 = N_2V_2$$

$$N_2 = N_1V_1/V_2$$

The normality of $KMnO_4$ (N_2) = ------ N

STANDARDISATION OF Fe^{2+} SOLUTION:

The Fe^{2+} Solution is given in a 100 ml standard flask .Make up the given Fe^{2+} solution up to the mark with distilled water and shake the flask well for uniform concentration. Pipette out 20 ml of Fe^{2+} Solution in to a250 ml conical flask add 20 ml of 2N sulphuric acid. Titrate the resulting solution with $KMnO_4$ taken in a burette. Pale pink colour is the end point. Repeat the titration to get concurrent values. Let the titer value be Y ml.

S.NO	Volume of Ferrous ammonium sulphate solution (ml)	Burette reading		Volume of $KMnO_4$ (ml)
		Initial	Final	
1	20	0	20	20
2	20	20	40	20
3	20	40	60	20

Titration -II

$N_3V_3 = N_4V_4$

Normality of $KMnO_4$ (N_3) = 0.1N

Volume of $KMnO_4$ (V_3) = ?

Volume of Fe^{2+} Solution (V_4) = 20ml

$N_3 = N_2V2/V_3$

The normality of Fe^{2+} (N_4) = ------N

The amount of Fe^{2+}present in given solution = N_4XEquivalent weight of Fe^{2+}

$=N_4X55.85=$----------g/l

RESULT:

1. Amount of Fe^{2+} iron present in the given solution =----------g/l
2. Normality of Fe^{2+} iron present in the given solution =----------N

VIVA QUESTIONS:

1) How to calculate equivalent weight of $KMnO_4$?
2) What is the structure of $KMnO_4$?
3) What is the oxidation state of Mn in reactant and Product sides?
4) Write the chemical equation of this experiment.
5) What is the purpose of using $KMnO_4$ on this experiment?
6) How to prepare 100ml of 0.1N $KMnO_4$ solution

Experiment – 4

CONDUCTOMETRIC TITRATION OF STRONG ACID Vs STRONG BASE (HCl vs NaOH)

AIM : To estimate the amount of HCl present in the given solution.

REAGENTS : Pure sample of oxalic acid, 0.02N HCl and 0.1 N NaOH.

APPARATUS: Conductivity meter, conductivity cell, burette, pipettes, beakers, stirrer.

PRINCIPLE :

Titration of strong acid HCl with strong base NaOH:

The rate of moment of charged particles is called as conductance or reciprocal of resistance. By using the conductance value to find out end point of titration is known as conductometry. Before base is added, the conductivity of acid solution is high (mainly due to the presence of highly mobile H+ ions). On gradual addition of NaOH from burette, highly mobile H+ ions of the acid are removed by the added OH- ions to form water molecules. Hence, the conductivity of the solution decreases progressively, till the end point is reached on further addition of NaOH, the conductivity of the solution will rise due to the addition of highly mobile OH- ions to the solution.

$$Hcl + NaOH \text{ ----------} \rightarrow NaCl + H_2O$$

PROCEDURE:

Preparation of standard oxalic acid solution:

Weigh out accurately about 0.63 g of pure crystalline oxalic acid into a clean 100 ml standard flask with the help of a funnel. Dissolve the crystals in a little distilled water and make up the solution to the mark by adding more distilled water. Stopper the flask and shake well to get uniform concentration of the solution. Calculate the normality of this prepared solution of oxalic acid.

CALCULATIONS:

Normality of oxalic acid N= (W/126)x10

Where W=Weight of oxalic acid, N = Equvalent weight of oxalic acid.

Standardization of the given NaOH solution :

Rinse a clean burette, first with distilled water and then with the given NaOH solution, and fill it up with the same solution. Rinse the pipette with the prepared oxalic acid solution and pipette out 20ml of it into a clean conical flask. Add about 2 to 3 drops of Phenolphthalein indicator. Titrate the solution against NaOH slowly and with constant shaking till the appearance of a permanent light pink colour. Note down the burette reading. Repeat the titrations till two concurrent readings are obtained.

REACTION:

$$\mathbf{H_2C_2O_4 + 2NaOH \longrightarrow Na_2C_2O_4 + 2H_2O}$$

S. No.	Volume of oxalic acid	Burette Reading		Volume of NaOH
		Initial	Final	
1	20 ml	0		

2	20 ml			

CALCULATIONS:

Titration -I

Normality of oxalic acid (N_1) = 0.1N

Normality of NaOH (N_2) = ?

Volume of oxalic acid (V_1) = 20ml

Volume of NaOH (V_2)= X

$$N_1V_1 = N_2V_2$$

$$N_2 = N_1V_1/V_2$$

The normality of NaOH (N_2) = ------ N

Conductometric titration of strong acid HCl with a strong base NaOH:

The burette is rinsed and filled with 0.1N NaOH. 20 ml of the given acid is placed in a 100 ml beaker. The cell washed with conductivity water is placed in HCl solution and is seen that the electrodes of the cell are completely immersed in the solution. The cell is fixed in a cell holder and is connected to the conductivity bridge. The conductance of HCl solution is noted. At a time 2 ml of NaOH from the burette is added into HCl solution. After each addition the solution is stirred gently with a glass rod and the conductance of HCl is noted. The titration is continued till 16 ml of NaOH is added.

MODEL GRAPH:

A graph of conductance against the volume of alkali added is plotted as shown in figure-3.1. Conductance curves are generally straight lines. The point of intersection of the two straight lines gives the volume of NaOH required for the complete neutralization of HCl

S.No.	Volume of NaOH in (ml)	Observed conductance in milli siemen (mhos)
1	0	32.4
2	2	30.25
3	4	28.3
4	6	26.1
5	8	24.1
6	10	22.5
7	12	20.2
8	14	18.3
9	16	16.4

10	18	14.3
11	20	9.8
12	22	11.4
13	24	13.2
14	26	14.8
15	28	15.6
16	30	16.1

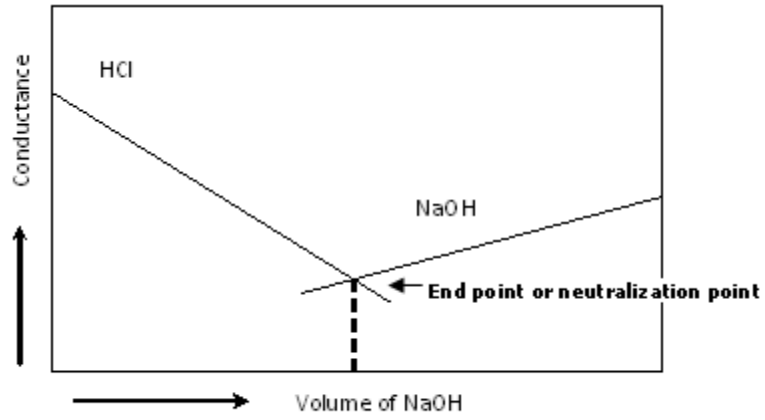


Figure-3.1

CALCULATIONS:

Titration -II

$N_1V_1 = N_2V_2$

Normality of NaOH (N_1) = 0.1N

Volume of NaOH (V_1) = ?

Volume of HCl Solution (V_2) = 20ml

The normality of HCl (N_2) = ------N

$N_2 = N_1V_1/V_2$

The amount of HCl present in given solution = N_4XEquivalent weight of HCl

$=N_2$ X 36.5=----------g/l

RESULT:

The normality of HCl present in the given solution is _____ N.

The amount of HCl present in the given solution is----------g/ml

VIVA QUESTIONS:

1. What is a Conductometric titration?
2. Explain the principle in this experiment?
3. What is the relationship between equivalent and molar conductance?
4. How to predict end point of titration?
5. What are the advantages of Conductometric titrations?
6. How to draw model graph?

Experiment -5

POTENTIOMETRIC TITRATION OF STRONG ACID Vs STRONG BASE (HCL vs NaOH)

AIM : To estimate the amount of HCl present in the given solution.

REAGENTS : Pure sample of oxalic acid, 0.1N HCl and 0.1N NaOH.

APPARATUS : Saturated calomel electrode, platinum electrode, quinhydrone electrode, potentiometer, salt bridge, burette, pipettes, beakers, stirrer.

PRINICPLE :

Quinhydrone (Q) is an equi molar mixture of Quinone $C_6H_4O_2$ and Hydroquinone $C_6H_4(OH)_2$ in acidic medium the following redox equilibrium is established.

$$Q+2H^+ + 2e^- = QH_2$$

A pinch of quinhydrone added to an acid solution and Pt wire or foil dipped in it constitutes a half-cell. Such electrodes are called indicator electrodes. This is coupled with a reference electrode through a salt bridge making it an electrochemical cell.

Ex: (-) saturated calomel electrode/Salt bridge/Quinhydrone/Acid solution/Pt(+)
(or)

(-) Pt, Hg/Hg_2Cl_2 (s) / KCl (saturated)/H^+ (?), QH_2/QPt(+)

Cell reaction:

$$2Hg(s)+Q+2Cl^- (aq)+2H^+ (aq) = Hg_2Cl_2 (s)+QH_2$$

Ecell = E right – E left (in terms of reduction potentials)

$$Ecell = EQE - 0.242 \quad \text{---------- (1)}$$

The left-hand half-cell is fixed i.e. its potential is constant. Now the right hand half-cell's potential depends on H^+ activity. The fundamental equation governing the effect of activity of the ions on the voltage of an electrode or a cell is Nernst equation given by

$$Ecell = E^0cell - (RT/nF) \ln K$$

Where K is equilibrium constant of a general reaction aA+bB+cC+dD
The emf is always balanced and the principle involved is Pogendorff's compensation principle.

The potential will be $E_{QE} = E^0cell - (RT/2F) \ln [QH2]/([Q][H^+]2)$
EQE = E0QE + 0.0591 log $[H^+]$ since $[QH_2]$= [Q] substituting in eqn 1. We have

$$Ecell = (E0QE + 0.0591 \log [H^+]) - 0.242$$

$$Ecell = 0.458 - 0.0591 \text{ PH i.e. Ecell is a function of PH}$$

During the titration, against an alkali, H^+ ion concentration will decrease. Correspondingly the Ecell decreases. The emf will change slowly in the beginning, however, at the end point relatively higher potential difference will be obtained and then again it will change slowly. After the sudden change also 5-6 more readings are taken. Repetition of the titration is done by adding 2 ml of NaOH at a time near the end point.

A graph is plotted between emf and volume of base added. The pint of inflexion where the curve changes its direction is the end point. A differential plot of $(\Delta E/\Delta V)$ against V gives a curve whose peak indicates equivalence point.

PROCEDURE:

Preparation of standard oxalic acid solution:

Weigh out accurately about 0.63 of pure crystalline oxalic acid into a clean 100 ml standard flask with the help of a funnel. Dissolve the crystals in a little distilled water and make up the solution to the mark by adding more distilled water. Stopper the flask and shake well to get uniform concentration of the solution. Calculate the normality of this prepared solution of oxalic acid.

CALCULATIONS:

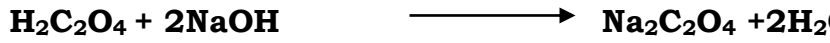
normality of oxalic acid N=(W/126)x10

Where W=Weight of oxalic acid, N=Equivalent weight of oxalic acid.

Standardization of the given NaOH solution:

Rinse a clean burette, first with distilled water and then with the given NaOH solution, and fill it up with the same solution. Rinse the pipette with the prepared oxalic acid solution and pipette out 20ml of it into a clean conical flask. Add about 2 to 3 drops of phenolphthalein indicator. Titrate the solution against NaOH slowly and with constant shaking till the appearance of a permanent light pink colour. Note down the burette reading. Repeat the titrations till two concurrent readings are obtained.

REACTION:



S. No.	Volume of oxalic acid	Burette Reading		Volume of NaOH
		Initial	Final	
1	20 ml	0	20	20
2	20 ml	20	40	20
3	20 ml	40	60	20

CALCULATIONS:

Titration -I

Normality of oxalic acid (N_1) = 0.1N

Normality of NaOH (N_2) = ?

Volume of oxalic acid (V_1) = 20ml

Volume of NaOH (V_2)= X

$$N_1V_1 = N_2V_2$$

$$N_2 = N_1V_1/V_2$$

The normality of NaOH (N_2) = ------ N

Potentiometric titration of strong acid HCl with a strong base NaOH:

20ml of the given acid is taken in 100ml beaker, and both platinum and calomel electrodes are placed in it. The ends of both the electrodes are connected to potentiometer. The cell Emf is recorded before the addition of base. NaOH solution from burette is added 2ml at a time. After each addition the solution is mixed gently and the EMF is noted.

From this titration, approximate value of NaOH required is found. The titration is repeated with adding lots of base at the beginning and 2 ml of base near the end point. The Ecell is plotted against the volume of NaOH. From the curve end point is noted. Another graph is obtained by plotting (ΔE/ΔV) against V (Average of two consecutive volumes of NaOH). From the graph equivalence point of titration is obtained as shown in figure 4.0 and figure-4.1

HCl Vs. NaOH

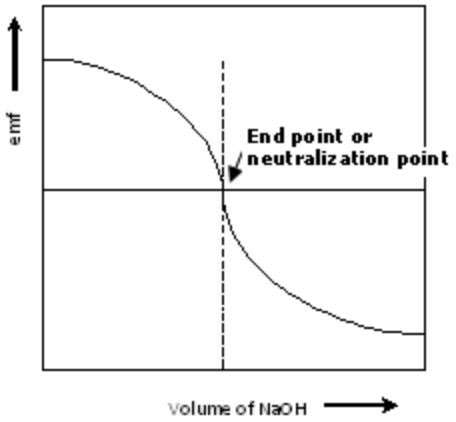


Figure-4.0.

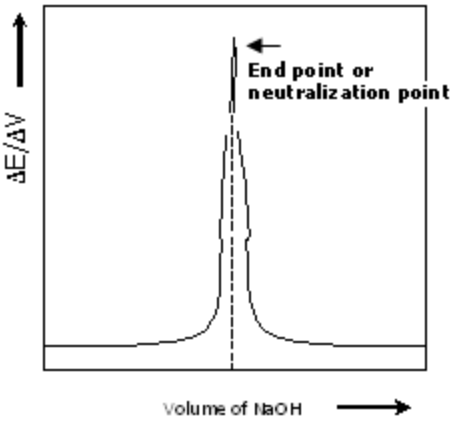


Figure-4.1.

OBSERVATION

S. NO.	Volume of NaOH in ml (V)	EMF (volts) (E)	ΔE(Difference between Two Consecutive E)	ΔV (Difference between two Consecutive V)	(ΔE/ΔV)
1	0			2	
2	2			2	
3	4			2	
4	6			2	

5	8			2	
6	10			2	
7	12			2	
8	14			2	
9	16			2	
10	18			2	
11	20			2	
12	22			2	
13	24			2	
14	26			2	
15	28			2	
16	30			2	

CALCULATIONS:

Titration -II

$N_3V_3 = N_4V_4$

Normality of NaOH (N_3) = 0.1N

Volume of NaOH (V_3) = ?

Volume of HCl Solution (V_4) = 20ml

The normality of HCl (N_4) = ------N

$N_4 = V_3N_3/V_4$

The amount of HCl present in given solution = N_4XEquivalent weight of HCl

$= N_4$ X 36.5=----------g/l

RESULT:

1. The normality of given HCl solution is N
2. The amount of HCl present in the given solution is _____g/l

VIVA QUESTIONS:

1. Define emf of a cell?
2. Define electrode potential?
3. Define a reference electrode?
4. Define reduction potential?
5. What is a calomel electrode?
6. What is the potential of hydrogen electrode?
7. What is standard electrode potential?

Experiment –6

Estimation of Fe^{2+} by Potentiometry using $K_2Cr_2O_7$

Aim: - To determine the **Fe^{2+}**by Potentiometryusing **$K_2Cr_2O_7$**

Apparatus: Burette, Burette stand, 20 ml pipette, 100 ml beaker and stirrer Potentiometer,Platinum electrode and calomel electrode.

Chemicals required:-

$Fe(NH_4)_2(SO_4)$ (Ferrous ammonium Sulphate (0.0125N) solution and Potassium Dichromate ($K_2Cr_2O_7$)(0.025N) solution

Principle:

The reference electrode used here is saturated calomel electrode (SCE). It consists of mercury metal covered with a paste of Hg + Hg_2Cl_2 ↓ in contact with saturated KCl solution and Pt Wire for electrical contact. The reduction potential of this electrode is 0.242V. This saturated calomel electrode functions as anode.

The **Indicator electrode** is a platinum electrode which responds rapidly to oxidation- reduction couples and senses the potential which depends upon the concentration ratio of the reactants & products of redox reactions. Here, the Pt electrode is in contact with a Ferrous-Ferric couple. This electrode functions as cathode.

Cell Representation: (-) Pt | Hg(1),Hg_2Cl_2(s)| KCl(sat)|| Fe^{3+},Fe^{2+}|Pt (+)

Cell Reaction: Anode:- 2 Hg + $2Cl^-$ → Hg_2Cl_2 + $2e^-$

Cathode:- $2Fe^{+3}$ + 2e- → $2Fe^{+2}$

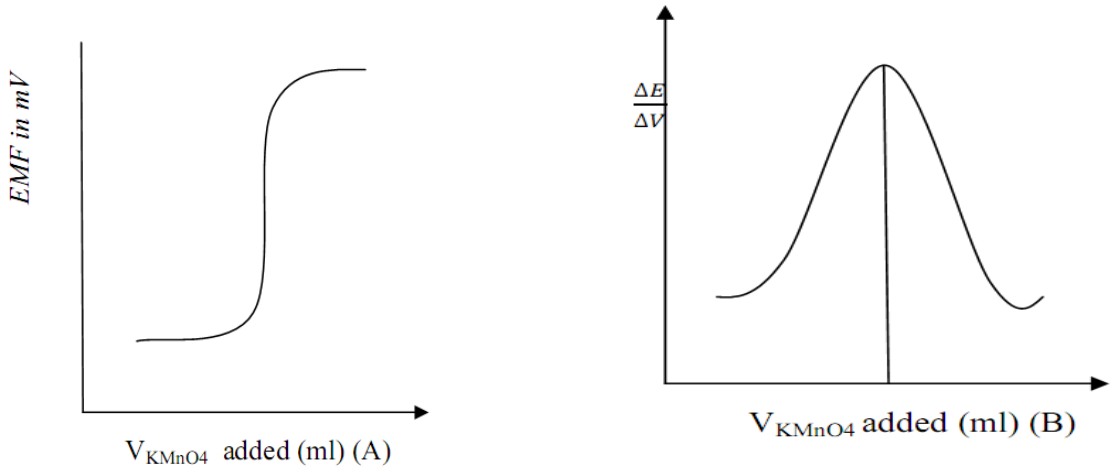
The cell potential is measured during the course of reaction and graphs are plotted. From the graphs end point of the titration is located and concentration is calculated.

Procedure:

Fill the burette with Standard$K_2Cr_2O_7$ solution and fix it to the stand. Pipette out 20 ml of test solution in a clean 100 ml beaker, place the platinum electrode and Saturated calomel electrode (SCE) in the solution, which creates a Fe^{+2}/Fe^{+3} couple. Connect the electrode to pH terminal. Switch on the instrument. Add Standard$K_2Cr_2O_7$ from burette in 2 ml portions to the ferrous solution, stir it and note the EMF. Continue the titration till a sudden inflextion in EMF occurs. Then take about 6 to 8 readings after inflexion in 2 ml INTERVALS. From the titrations approximate volume of $K_2Cr_2O_7$ required is found out.The titration is repeated with addition of $K_2Cr_2O_7$ in 0.1 ml lots in the vicinity of end point.

Graph:-

1. Plot a graph between volume of **$K_2Cr_2O_7$ added on X-axis** and **emf on Y-axis** .Then we get the following curve.



2. Plot a graph between ΔE/Δv values against volume of $K_2Cr_2O_7$ added. The maximum of the curve represents the end point.

Tabular form:-

S.No	Volume of $K_2Cr_2O_7$ added(ml)	Emf	ΔE	ΔV	ΔE/ ΔV
1	0 ml			2	
2	2 ml			2	
3	4 ml			2	
4	6 ml			2	
5	8 ml			2	
6	10 ml			2	
7	12ml			2	
8	14ml			2	
9	16ml			2	
10	18ml			2	
11	20 ml			2	
12	22 ml			2	
13	24 ml			2	
14	26 ml			2	
15	28 ml			2	
16	30 ml			2	

Calculation:-

$N_1V_1 = N_2V_2$

Normality of $K_2Cr_2O_7$ (N_1) = 0.1N

Volume of $K_2Cr_2O_7$ (V_1) = by graph

Volume of Fe^{2+} Solution (V_2) = 20ml

The normality of Fe^{2+} (N_2) = ?

$$N_2 = N_1V_1/V_2$$

The amount of Fe^{2+} present in given solution = N_2XEquivalent weight of Fe^{2+}

$=N_2X55.85=$----------g/l

RESULT:

1. Amount of Fe^{2+} iron present in the given solution =----------g/l
2. Normality of Fe^{2+} iron present in the given solution =----------N

VIVA QUESTIONS:

1. Define emf of a cell?
2. Define electrode potential?
3. Define a reference electrode?
4. Define reduction potential?
5. What is a calomel electrode?
6. What is the potential of hydrogen electrode?
7. what is differential curve?

Experiment –7

PREPARATION OF POLYMER – BAKELITE

AIM: To prepare phenol formaldehyde resin (Bakelite).

CHEMICALS: Glacial acetic acid, 40% formaldehyde solution, Phenol, conc. H_2SO_4

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

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

THERMOSETS: The polymers which on heating change irreversibly into hard rigid and infusible materials are called thermosetting polymers. These polymers are usually prepared by heating relatively low molecular mass, semi fluid polymers, which becomes infusible and form an insoluble hard mass on heating. The hardening on heating is due to the formation of extensive cross-linking between different polymeric chains. This lead to the formation of a 3-Dimensional network of bonds connecting the polymer chains. Since the 3D network structure is rigid and does not soften on heating, the thermosetting polymers cannot be reprocessed. Some important examples of thermosetting polymers are Urea-Formaldehyde resin and Melamine-Formaldehyde resins.

PROPERTIES:

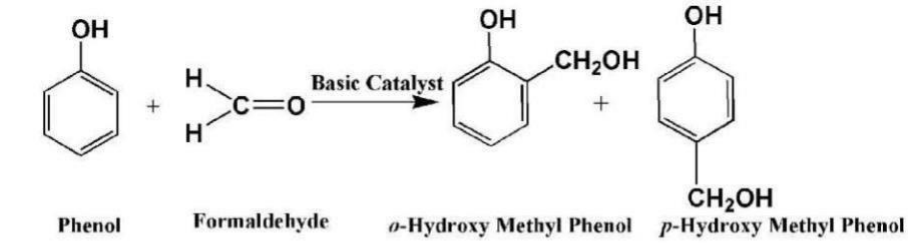
1. Phenol- formaldehyde resins having low degree of polymerization are soft. They possess excellent adhesive properties and are usually used as bonding glue for laminated wooden planks and in varnishes and lacquers.
2. Phenol- formaldehyde resins having high degree of polymerization are hard, rigid, scratch resistant and infusible. They are resistant to non-oxidizing acids, salts and many organic solvents. They can withstand very high temperatures. They act as excellent electrical insulators also.

Uses:

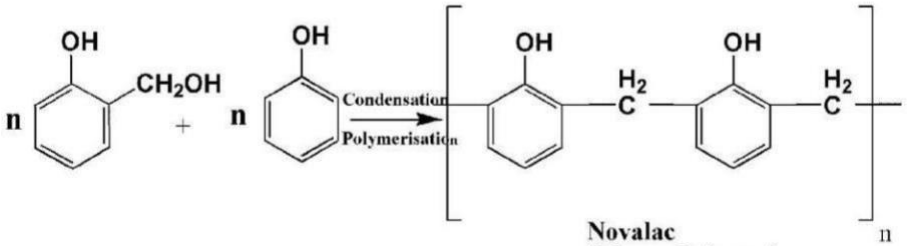
- They are used for making molded articles such as radio and TV parts, combs, fountain pen barrels, phonograph records etc.
- They are used for making decorative laminates, wall coverings etc.
- They are used for making electrical goods such as switches, plugs etc.
- They are used for impregnating fabrics wood and paper.
- They are used as binding glue for laminated wooden planks and in varnishes and Lacquers.
- Sulphonated phenol-formaldehyde resins are use as ion-exchange resins.

PREPARATION: PFs are prepared by reaction of phenol with formaldehyde in the presence of acidic or basic catalyst. The process may be carried out as follows:

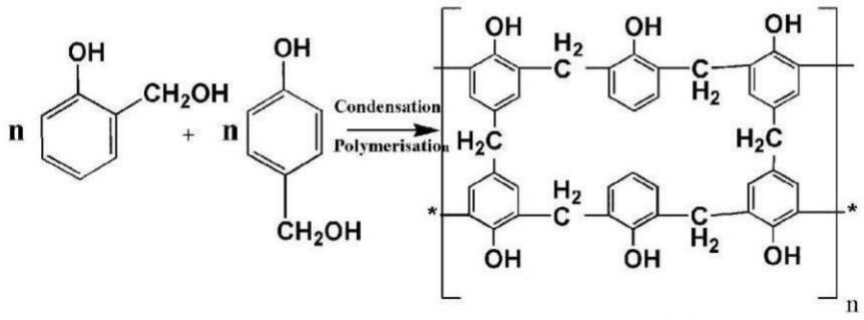
A mixture of phenol and formaldehyde are allowed to react in the presence of a catalyst. The process involves formation of methylene bridges in *ortho*, *para* or *both ortho* and *para* positions. This results first in the formation of linear polymer (Called NOVALAC) and then in to cross- linked polymer called phenol-formaldehyde resin or *Bakelite.*



Phenol **Formaldehyde** ***o*-Hydroxy Methyl Phenol** ***p*-Hydroxy Methyl Phenol**



Novalac
(Linear Polymer)



Bakelite
(Cross-linked Polymer)

PROCEDURE:

1. Place 5ml of glacial acetic acid and 2.5ml of 40% formaldehyde solution in a 500ml beaker and add 2 grams of phenol.
2. Add few ml of conc. sulphuric acid into the mixture carefully. Within 5 min. a large mass of plastic is formed.
3. The residue obtained is washed several times with distilled water, and filtered product is dried and yield is calculated.

RESULT: The weight of the phenol formaldehyde resin is — g.

PRECAUTIONS*:*

- The reaction is sometimes vigorous and it is better to be a few feet away from the beaker while adding the H2SO4 and until the reaction is complete.
- The experiment should be preferably carried out in fume cupboard.

VIVA QUESTION:

1. What is the other name given for Phenol formaldehyde? Phenol What are the monomers used for preparation of Bakelite?

Give main uses of the phenol formaldehyde resin.

2. What type of co-polymer is phenol formaldehyde resin? It is a thermosetting polymer?

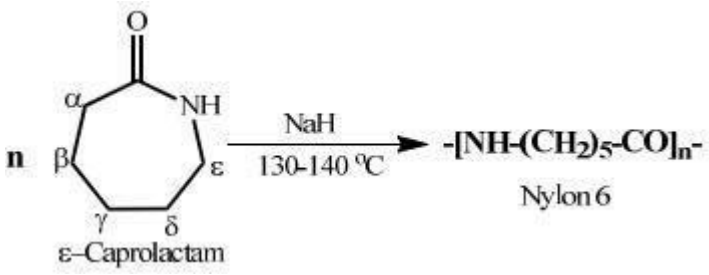
3. What are thermosetting polymers?

4. Briefly describe the properties of phenol resins?

Experiment –8

PREPARATION OF NYLON 6

AIM: To prepare a pure organic polymer Nylon -6
APPARATUS: Beaker (100ml), Measuring jar, Plastic or silicone spatula, Weighing balance, Magnetic stirrer, Glass rod, round bottom flask, Condenser
CHEMICALS REQUIRED: Caprolactam, Dilute sulphuric acid (4 ml), Water
PRINCIPLE: Nylon 6 is produced by ring-opening chain growth polymerization of caprolactam in the presence of water vapor and an acid catalyst at the melt. After removal of water and acid, the nylon 6 is melt spun at 250°–260°C into fibers. When epsilon (ε)-caprolactam is heated with water at high temperature it undergoes ring opening polymerization to form the polyamide polymer called nylon-6.



The name nylon-6 is given on the basis of six carbon atoms present in the monomer unit. Nylon-6 has high tensile strength and luster, nylon-6 fibres are used for manufacture of tyre cords, fabrics and ropes.

PROCEDURE:

1. Take 5 gm of caprolactam in the test tube containing a side arm which has been previously flushed with N2.
2. Maintain inert atmosphere in the tube during the experiment by passing small stream of nitrogen through side arm.
3. Insert the thermometer to check the temperature and heat the test tube at 135-140°C to melt the caprolactam.
4. Add NaH (0.1 gm) and dissolve it to form the sodium derivative of caprolactam.
5. Add N-acetyl caprolactam (0.7 gm) at the same temperature.
6. After several minutes remove thermometer.
7. Mix the contents by shaking and mildly heating the test tube for almost 10 minutes. After 5-10 minutes the solid product will form which indicate the completion of the reaction

8.Transfer the product in the Petri-dish and cool it to get the solid Nylon 6.

9. The melting point of Nylon 6 is 200-220 C.

VIVA VOCE:

1. Name the principle involved in the preparation of Nylon -6.

2. Give the other name of polyoxyethylene.

3. Give the uses of Nylon -6

4. Who developed Nylon - 6?

5. What are the important properties of Nylon – 6?

6. Which class of polymer does Nylon – 6 belong to?

Experiment – 9

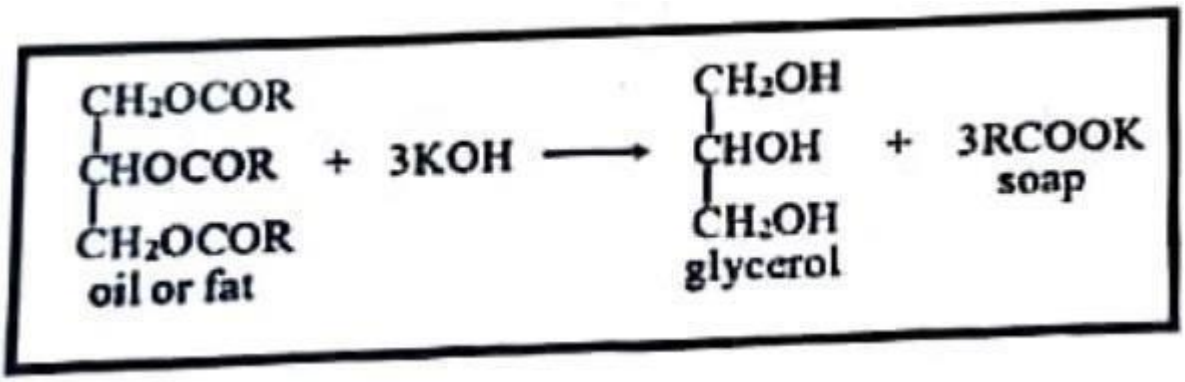
ESTIMATION OF ACID VALUE OF GIVEN LUBRICANT OIL

AIM: To estimate the Acid value of given oil/lubricant oil.

APPARATUS: 100 ml conical flask, Burette, Pipette, Beaker, Dropper.

CHEMICALS: Ethanol, 0.1 N potassium hydroxide (KOH), Phenolphthalein.

PRINCIPLE: The milligrams of KOH required to completely neutralize tree acid present in 1 gm of oil is known as acid value or neutralization number. The acid value is determined by directly titrating the oil/'fat in an alcoholic medium against standard potassium hydroxide solution.



Reagents:

1) Phenolphthalein indicator solution: Dissolve 1 gram of phenolphthalein in 100ml of ethyl alcohol.
2) Ethyl alcohol: 95% percent alcohol or rectified spirit neutral to phenolphthalein indicator.
3) Standard Potassium hydroxide (0.1 N): The solution should be colourless and stored in a brown glass bottle.

Procedure:

Weigh accurately about 10gms of oil and transfer it into a 250 ml conical flask. Add 50 ml of freshly neutralized hot ethyl alcohol and about one ml of phenolphthalein indicator solution. Heat the mixture for about fifteen minutes on water bath (75-80°C) Titrate the hot solution against standard

KOH Solution, while shaking the contents vigorously. The end point is the appearance of light pink colour which should persist at least for 15 seconds. The weight of the oil/fat taken for the estimation and the strength of the alkali used for titration shall be such that the volume of alkali required for the titration does not exceed l0ml.

Observation:

No. of Observation	Initial Burette Reading (ml)	Final Burette Reading (ml)	Difference (ml)	Average (ml)
1				
2				
3				

Calculation:

$$\text{Acid value} = \frac{\text{Vol. in KOH consumed}}{\text{Weight of oil taken in gram}} \times 5.6$$

RESULT : Acid value of the given oil is = mg of KOH/1g

Viva Questions:

1. What is an acid value?
2. Acid value is also called as?
3. What does the acid value indicate?
4. What is significance of determining the acid value of an oil?
5. What is the Analytical importance of acid value?
6. Why do we need acid value?
7. What does high acid value mean?

Experiment – 10

DETERMINATION OF VISCOSITY

AIM : To estimate the viscosity of given organic liquid by using Ostwald's viscometer.

APPARTUS : Ostwald's viscometer, 10ml pipette, volumetric flask, 100ml beaker, rubber wok, stop clock

CHEMICALS : 1% ethanol, 1% chloroform, 1% acetic acid.

PRINCIPLE : Viscosity is the property of fluid which determines the rate of flow, the liquid molecules in the tube experience internal resistance to flow the relative motion of adjacent layers of liquid is opposed by internal friction the internal resistance is known as viscosity of liquid.

Coefficient of viscosity is determined as the force that must be exerted b/w two parallel layers of unit area & unit distance apart. In order to maintain the unit velocity, streaming of one layer apart the other. Ostwald derived an equation for determination of viscosity as follows:

$$\eta = \frac{4r^4tp}{8\,vl}$$

Where,

r = Radius of viscometer rube

p = Pressure

v = Volume

l = Length of the tube

for equal volumes of liquid same equipment the equation can be reduced as

$$\frac{\eta_1}{\eta_2} = \frac{p_1t_1}{p_2t_2}$$

Where

p = Hydrostatic Pressure

t = Temperature

In terms of density,

$$\frac{\eta_1}{\eta_2} = \frac{d_1 t_1}{d_2 t_2}$$

Where,

$\eta 1$ = Viscosity of water (8.90 milli poise)
$\eta 2$ = Viscosity of organic liquid
d_1 = density of water
d_2 = density of organic liquid
t_1 = time taken for water
t_2 = time taken for organic liquid

PROCEDURE:

The Ostwald's viscometer consists of a fine capillary tube with a bulb A at its upper end & in 'v' shape. Bulb'B' at its lower end. To marks x & y are made on the tube above & below A. clean the viscometer with distilled water set the viscometer & introduce a volume of given organic liquid in the bulb A up to the mark x. Now allow the liquid to flow freely through the capillary & the start the stop clock & note the time (taken) which is required for flow the liquid from mark x to mark y. Repeat the same procedure for concurrent readings by taking exactly the same volume of liquid. By knowing the density of organic liquid we can calculate the viscosity of the given liquid.

S. No.	**Name the organic Liquids**	**Taken time flow of liquid x to y mark**		**Average**	**Viscosity (mille poise)**
		Expt-1	**Expt-2**		
1	**H_2O**				
2	**1% ethanol**				
3	**1% chloroform**				
4	**1% acetic acid**				

RESULT: viscosity of 1% ethanol = _____ millipoises
viscosity of 1% chloroform = _____ millipoises
viscosity of 1% acetic acid = _____ millipoises

VIVA QUESTIONS:

1. Define viscosity?
2. Mention different types of viscometers?
3. What is the principle involved in the determination of viscosity?
4. Give the equation for the determination of viscosity?
5. What is the effect of temp on viscosity?
6. What is the value of viscosity of water?