INDEX

Sl. No	Date	Title of the Experiment	Signature of the Staff	Remarks
		Instrumentation Experiments		
1A		ESTIMATION OF COPPER PRESENT IN E-WASTE BY OPTICAL SENSOR **(COLORIMETRY)**		
1B		ESTIMATION OF % OF IRON IN STEEL INDUSTRY EFFLUENT BY USING OPTICAL SENSOR (COLORIMETRY) (CIVIL)		
2		ESTIMATION OF ACID MIXTURE BY **CONDUCTOMETRIC SENSOR**		
3		DETERMINATION OF pKa OF VINEGAR USING pH SENSOR (Glass electrode)		
4		ESTIMATION OF IRON IN RUST SAMPLE BY **POTENTIOMETRIC SENSOR**		
5		DETERMINATION OF VISCOSITY COEFFICIENT OF CONDUCTIVE INKS(ANY LIQUID)		
		Volumetric Experiments		
1		ESTIMATION OF **TOTAL HARDNESS** OF WATER BY EDTA METHOD		
2		DETERMINATION OF **CHEMICAL OXYGEN DEMAND** OF INDUSTRIAL WASTE WATER SAMPLE		
3		DETERMINATION OF TOTAL ALKALINITY OF GIVEN WATER SAMPLE		
4		ESTIMATION OF IRON IN TMT BAR STEEL BY DIPHENYL AMINE INDICATOR METHOD		
5A		DETERMINATION OF HYDROGEN PEROXIDE IN PCB CLEANER (PERMANGANATE METHOD) (ECE&EEE)		
5B		DETERMINATION OF ACID VALUE OF BIO FUEL (ME STREAM)		
5C		DETERMINATION OF PERCENTAGE OF CaO IN CEMENT BY COMPLEXOMETRIC METHOD (CV STREAM)		

General Instructions

- **Keep your working table clean and tidy**
- **Keep your apparatus clean**
- **Keep the reagents bottles in their proper place after use. Do not alter their order. Do not contaminate the reagents.**
- **Keep gas taps and taps closed when not in use.**
- **Use candle to light burner. Do not use paper torches for the purpose.**
- **Do not throw any waste paper in the sink. Throw them into the dustbin provided.**
- **Do not pour concentrated acids in to the sink. If they are to be poured, flush them using water liberally.**
- **To turn to be a better analyst, understand the theory behind the reaction you carryout.**
- **Record tour observations as and when you proceed and not after completion.**
- **Use the specified quantities of reagents.**
- **Do not walk with barefooted in the laboratory.**
- **Do not use wet hand during weighing.**

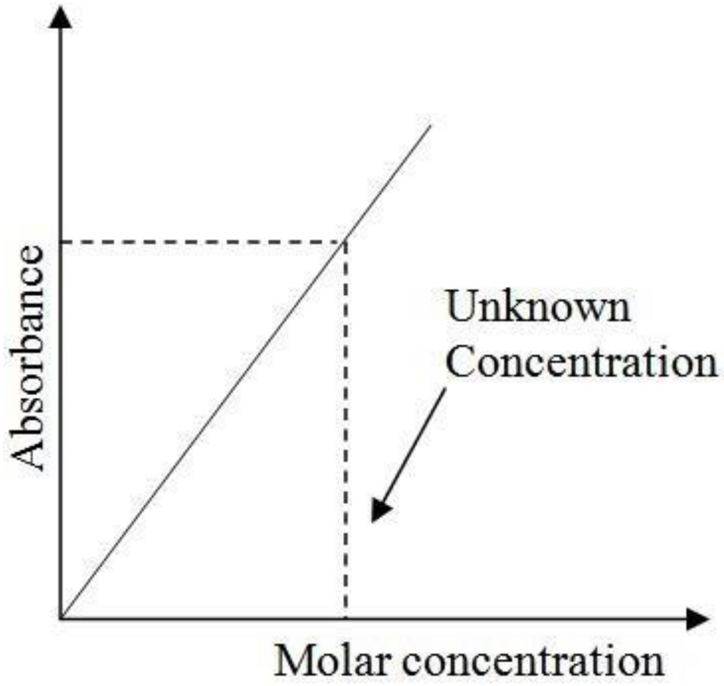
PART-A

Instrumentation Experiments

(COLORIMETRY)

1A & 1B

$$Cu^{+2} + 4NH_3 \longrightarrow [Cu(NH_3)_4]^{2+}$$



1A ESTIMATION OF COPPER PRESENT IN E-WASTE BY OPTICAL SENSOR (COLORIMETRY)

Experiment No: **Date:**

EXPERIMENT: Estimation of copper colorimetrically

REQUIREMENT: Photoelectric colorimeter, Nessler's tubes, 100ml volumetric flasks numbered 1 to 6

CHEMICALS: 0.016 / 0.032 M $CuSO_4$ solutions, NH_4OH (1:1)

PRINCIPLE: Colorimetric analysis depends upon the measurement of quantity of light absorbed by a coloured solution. Quantitative analysis by colorimetry is based on Beer-Lambert's law which can be expressed by the relation

$$\mathbf{Log\ (I_o/I_t) = \varepsilon C x}$$

where I_o = Intensity of the incident light

I_t= Intensity of the transmitted light

C = Concentration of the coloured constituent in the solution

x = Thickness of the coloured medium (path length)

ε =Molar absorption coefficient

The term log (I_o/I_t) is called the absorbance or optical density of light absorbing medium, since ε is a constant and if thickness is kept constant, absorbance changes linearly with concentration within a specific concentration range.

Cupric ions react with ammonia to form deep blue color due to the formation of cupric ammonium complex ion.

A series of standard solutions containing cupric ions treated with ammonia and the absorbance of each of these solutions is measured at 620nm (λmax at which the solution shows maximum absorbance) A calibration curve is obtained by plotting molar concentration against absorbance.A know volume of the sample solution is also treated with ammonia in the way as the standards and its absorbance is measured. Its concentration is determined from the calibration curve.

TABULAR COLUMN:

V. F No.	Molar Concentration	Absorbance or Optical Density
1		
2		
3		
4		
Test Solution	**Unknown (M)**	

Molar Concentration of Test solution from Graph = M = ________________

CALCULATIONS:

Amount of copper present in the test solution = M X Eq. Wt of Cu

= M X 63.55 g/L

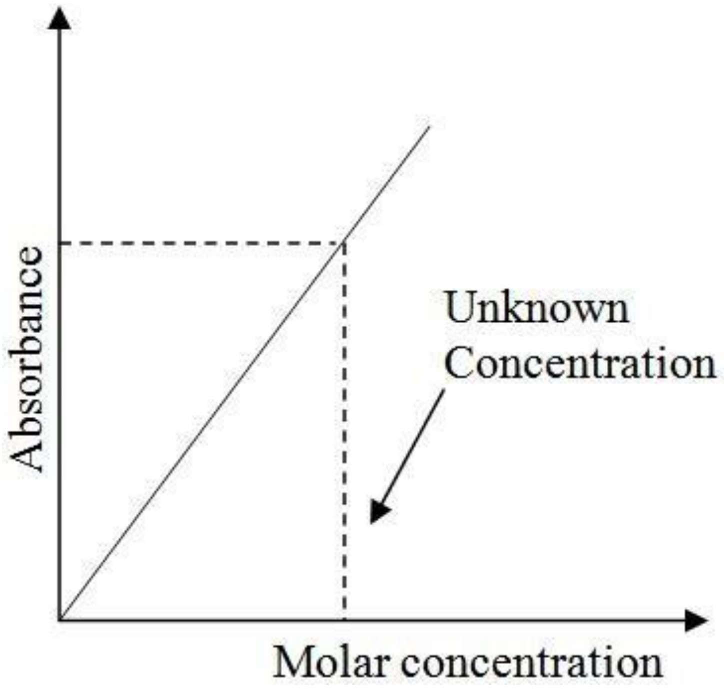
=___________g/L

PROCEDURE:

Transfer 5, 10, 15 and 20ml of the copper sulphate solution into separate labelled 100ml volumetric flasks (using burette) .Add 5ml of ammonium hydroxide solution of each of them (Using measuring jar) and make up to the mark with distilled water and mix well. To the test solution taken in 100ml volumetric flasks, add 5ml of NH_4OH and make upto the mark and mix Well. Prepare a blank solution by diluting 5ml of ammonium hydroxide in 100ml volumetric flasks up to the mark with distilled water and mix well. After 10 minutes, measure the absorbance (O.D) of the solution against blank solution at 620nm (use suitable filter) using colorimeter.

Draw a calibration curve by plotting molar concentration of copper sulphate against absorbance, using calibration curve determine the molar concentration of the test solution and calculate the amount of copper.

RESULT: Amount of copper in the given test solution =________g/L


Absorbance
Unknown
Concentration
Molar concentration

1B ESTIMATION OF % OF IRON IN STEEL INDUSTRY EFFLUENT BY USING OPTICAL SENSOR (COLORIMETRY) (CIVIL)

Experiment No: **Date:**

AIM: To estimate colorimetrically the amount of iron in the given sample

PRINCIPLE:

Traces of iron are usually determined by colorimetrically with thiocynate as reagent, ferric ion reacts with thiocynate to give an intensely red coloured compound. Depending upon the concentration of thiocynate a series of complexes of red colour are formed Therefore in colorimetric determination of iron, it is advisable to use excess of reagent. H_2SO_4 should not be used to prevent the hydrolysis of iron. Because the SO_4^{2-} ions have the tendency to form complex with ferric ion (Fe^{+3}) and thus intensity decreases with increase in concentrated H_2SO_4

$$6NH_4SCN + Fe^{3+} \longrightarrow (NH_4)_3[Fe(SCN)_6] \text{ (RED COLOUR COMPLEX) } + 3NH_4^+$$

The absorbance of the above red colour complex is measured around 480nm.

TABULAR COLUMN:

V. F No.	Molar Concentration	Absorbance or Optical Density
1		
2		
3		
4		
Test Solution	Unknown (M)	

Molar concentration of Test solution from graph = M = ___________

CALCULATION:

Amount of iron present in the test solution = M × Eq. Wt of Fe^{+3}

= M × 55.84

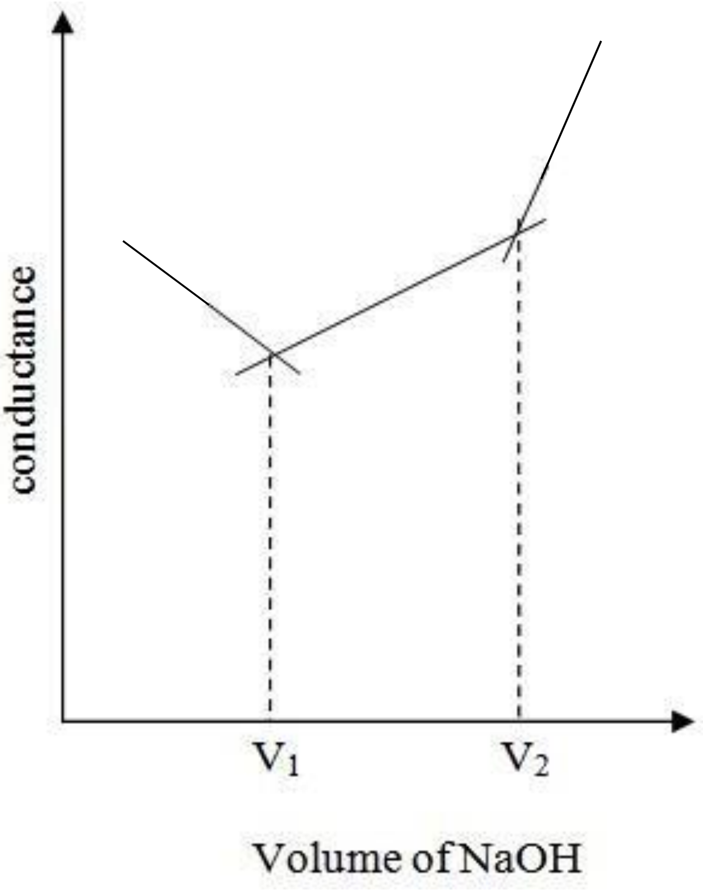
PROCEDURE:

0.5, 1.0, 1.5 and 2.0mL of the standard ferric ion solution (0.5mg/ml) are pipetted into different 50ml standard flask. To each 2.5ml of 4N HCl and 5ml of 20% NH_4SCN are added and the solution in each flask is made up to mark using distilled water. A blank is prepared in a similar way except adding the standard ferric solution. The intensities of colour developed are compared by using colorimeter at 480nm. To the given test solution 2.5ml of 4N HCl and 5ml of 20% NH_4SCN are added and made upto 50ml. The optical density of each solution is measured using a colorimeter against a blank solution at 480nm. A standard calibration curve is drawn by plotting the concentration on X-axis against absorbance on Y-axis. A straight line is obtained and from this the concentration of unknown can be determined.

RESULT: The amount of iron in the given test solution= __________ g/l

CONDUCTOMETRIC SENSOR

$$\mathbf{NaOH + HCl \longrightarrow NaCl + H_2O}$$



2 . ESTIMATION OF ACID MIXTURE BY CONDUCTOMETRIC SENSOR

Experiment No: **Date:**

EXPERIMENT: Estimation of acids in acid mixture conductometrically

REQUIREMENT: Conductometer , conductivity cell, 100ml Beaker, Glass rod , Micro burette 10ml

CHEMICALS: Acid Mixture ($HCl + CH_3COOH$) and 1N NaOH

PRINCIPLE: In Conductometeric titrations, there is a sudden change in conductance of the solution near the neutralization point. However, change is not sharp and hence the neutralization point is determined graphically by plotting conductivity against titrated values. The principle underlying conductometric titrations is the replacement of ions of a particular conductivity by ions of different conductivity during the titration

In the titration of a mixture of weak acids and strong acid against a strong base, conductance initially decreases upon adding NaOH to acid mixture owing to the substitution of highly mobile (H^+) ion by the less mobile Na^+ion. This trend continues till all the hydrogen H^+ ions of HCl are replaced (i.e. , the strong acid is neutralized), continued addition of NaOH raises the conductance moderately, as the weak acid CH_3COOH is converted into its salt, CH_3COONa further addition of NaOH raises the conductance steeply due to the presence of free OH^- ions.

PROCEDURE: Transfer 50ml of the given acid mixture into a beaker; fill a 10ml microburette with standard 1N NaOH solution. Place the conductivity cell inside the beaker so the electrodes are immersed completely in the mixture. Connect the cell to the conductivity bridge and measure the conductance.

Add 0.5ml NaOH from the burette, mix the solution well and measure the conductance of solution Continue the measurement of conductance after addition of 0.5 ml NaOH, every time . Take 20 such readings. Plot the graph, volume of NaOH against conductance.

TABULAR COLUMN:

Volume of Na OH in ml	Conductance
0.5	
1.0	
1.5	
2.0	
2.5	
3.0	
3.5	
4.0	
4.5	
5.0	
5.5	
6.0	
6.5	
7.0	
7.5	
8.0	
8.5	
9.0	
9.5	
10.0	

CALCULATIONS:

Volume of NaOH required for the neutralization of HCl (from graph) = V_1 =__________ml

V_2 =__________ml

Volume of NaOH required for the neutralization of CH_3COOH = $V_2 - V_1$ =__________ml

1. a) Normality of HCl = $\frac{1 \times V_1}{50}$ = ____________N

   b) Amount of HCl = Normality X Eq. Wt of HCl

   = Normality X 36.5 g /l = ____X 36.5 g /l = __________g /l

2. a) Normality of CH_3COOH = $\frac{1 \times (V_2 - V_1)}{50}$ =__________N

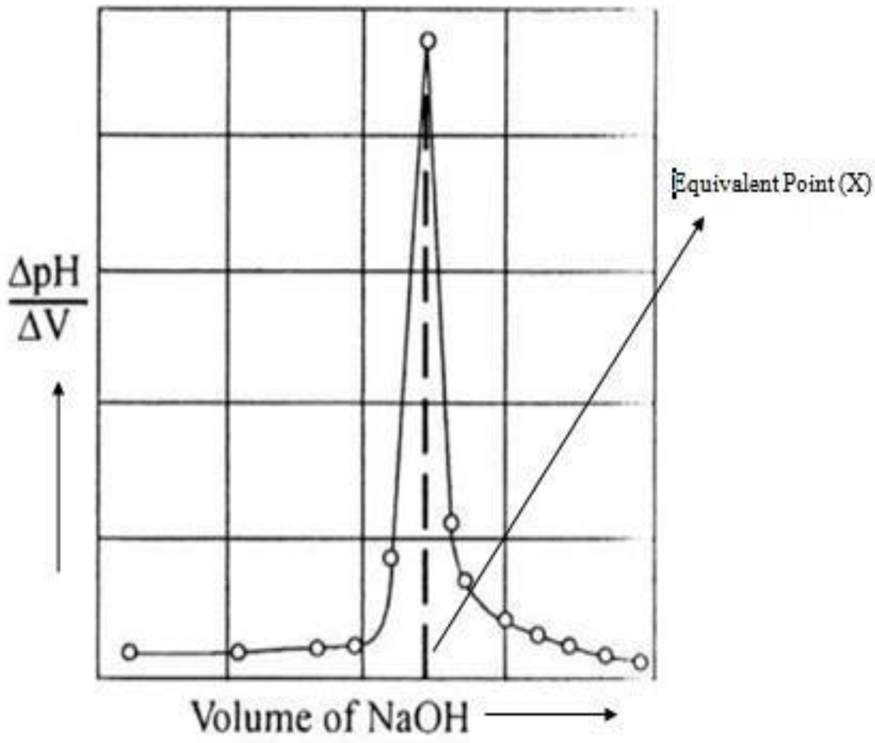
   b) Amount of CH_3COOH = Normality X Eq. Wt of CH_3COOH

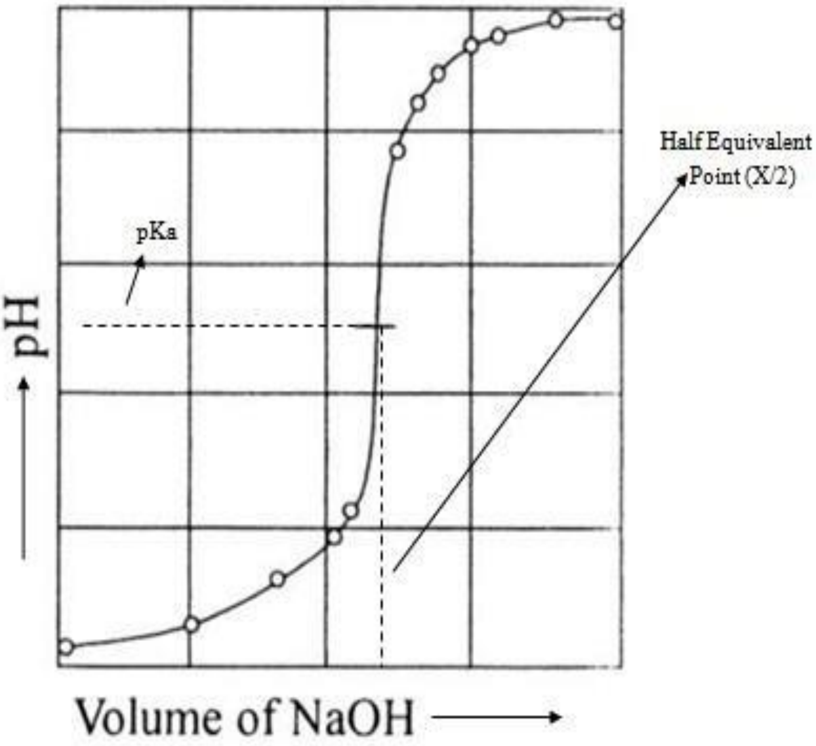
   = Normality X 60 g /l= ____X 60 g /l = ___________ g /l

RESULT:

1. Amount of HCl present in the given acid mixture =___________g/l
2. Amount of CH_3COOH present in the given acid mixture =___________g/l

pH METER


Equivalent Point (X)
$\frac{\Delta pH}{\Delta V}$
Volume of NaOH


Half Equivalent Point (X/2)
pKa
pH
Volume of NaOH

3. DETERMINATION OF pKa OF VINEGAR USING pH SENSOR (Glass electrode)

Experiment No: **Date:**

EXPERIMENT: Determination of pKa value of given weak acid using pH meter

REQUIREMENT: pH-meter, glass electrode - calomel electrode assembly, 50ml beaker, glass rod

CHEMICALS: NaOH, Weak Acid

PRINCIPLE: During the titration of an acid with base, pH of the solution rises gradually at first, then more rapidly, until at the equivalence point, there is a very sharp increase in pH for a very small quantity of added base. Once the equivalence point crosses, the pH increases slightly on addition of excess base. The titration curve is obtained by plotting changes in pH at different amount of the base added as shown in the graph.

PROCEDURE: Transfer 25ml of weak acid into a beaker using pipette. Immerse a glass electrode -calomel electrode assembly into the acid and connect the cell to pH-meter. Measure the pH of the acid. Fill a burette with NaOH. In the beginning add large increments (say 0.5ml) of NaOH to the weak acid stir the solution carefully and measure the pH after each addition. When the pH begins to show a tendency to increase rapidly add small increments (say 0.1ml) of NaOH and measure the pH after each addition. Continue till there is slight increase in pH on the addition of NaOH. Tabulate the readings.

Plot a graph of pH against V and determine the equivalence point. (X) Determine the value of pKa as pH at half equivalence point(X/2)

TABULAR COLUMN:

Volume of NaOH (V)	pH	ΔpH	ΔV	ΔpH/ΔV

CALCULATIONS:

According to Hendersen – Hasselbach equation

$$\mathrm{p}H = \mathrm{p}K\mathrm{a} + \log \frac{[\mathrm{sal}t]}{[acid]}$$

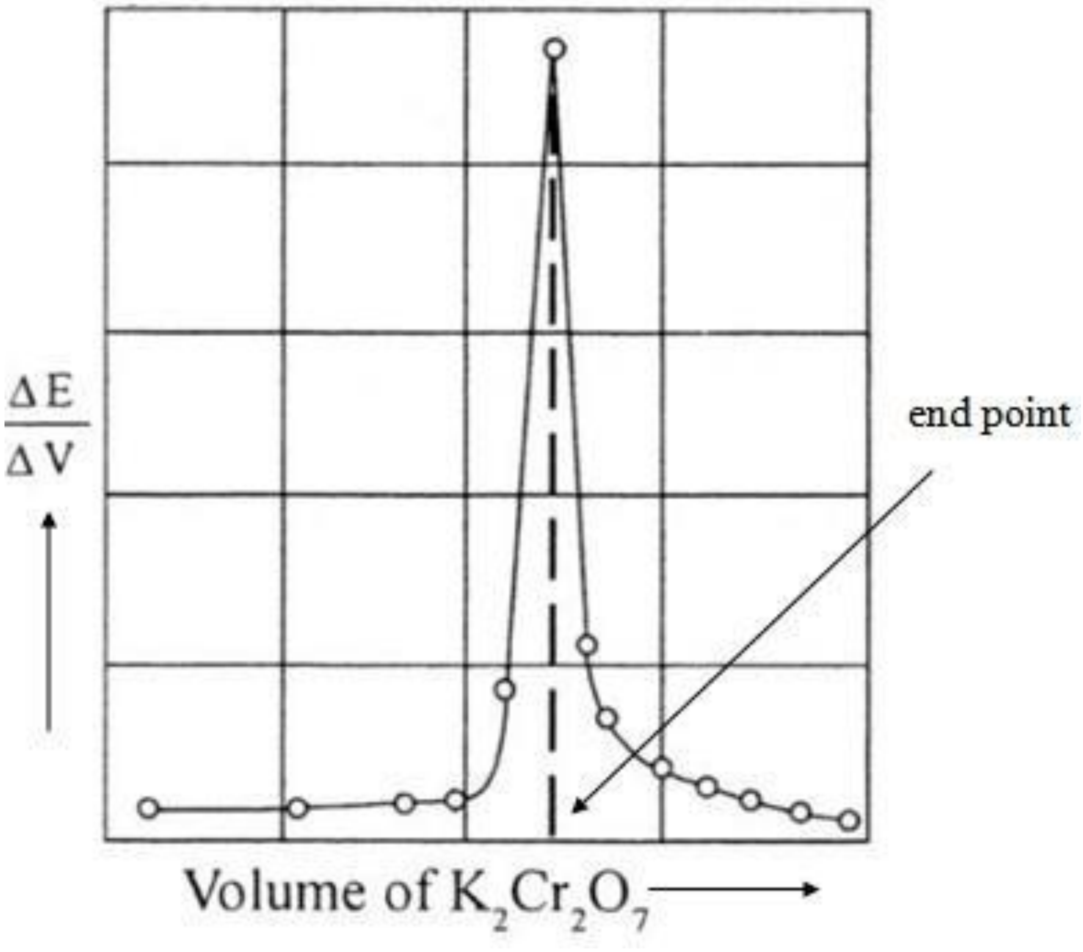
Where Ka is dissociation constant of the acid and pKa= log Ka

At half equivalence point, [salt] = [acid] and hence pH=pKa

Therefore, pH at half equivalence point gives pKa of weak acid

RESULT: The pKa value of given weak acid = _______

POTENTIOMETRY


$\frac{\Delta E}{\Delta V}$
end point
Volume of $K_2Cr_2O_7$

4. ESTIMATION OF IRON IN RUST SAMPLE BY POTENTIOMETRIC SENSOR

Experiment No: **Date:**

EXPERIMENT: Estimation of FAS potentiometrically using standard potassium dichromate solution

REQUIREMENT: Potentiometer (pH-meter, Plantinum electrode and calomel electrode) beaker, burette and glass rod.

CHEMICALS: FAS solution, dil.H_2SO_4, 0.5N $K_2Cr_2O_7$ solution

PRINCIPLE: Redox titrations can be carried out potentiometrically using platinum-calomel electrode combination in a manner similar to acid-base neutralizations.

For the reaction

$$\text{Reduced Form} \longrightarrow \text{Oxidised Form} + n \text{ electrons}$$

Potential is given by Nernst Equation

$$E = E_0 + \frac{0.0591}{n} \log \frac{[oxidised\ form]}{[reduced\ form]}$$

Where 'E^o'is the standard electrode potential of the system. The potential of the system is thus controlled by the ratio of concentration of the oxidized to that of the reduced species present. AS the reaction proceeds, the ratio alters and hence the potential changes more rapidly in the vicinity of the end point of the titration. This may be followed potentiometrically and a plot of change in a potential against volume (titration curve) is characterized by a sudden change in the potential at the equivalence point.

PROCEDURE:

Transfer 25ml of FAS solution into a beaker using pipette. Add 2 test-tube full of dilute sulphuric acid. Immerse the electrode assembly into the solution in the beaker and connect the electrodes to a potentiometer and measure the potential. Fill the burette with $K_2Cr_2O_7$ add 0.5 ml of $K_2Cr_2O_7$ solution to the beaker stir the solution carefully and measure the potential after 15 seconds. Continue the procedure till potential shows tendency to increase rapidly. Now add $K_2Cr_2O_7$ solution in increments of 0.1 ml and measure the potential after each addition. Determine the end point by differential method i.e., by plotting $\Delta E/\Delta V$ against volume of $K_2Cr_2O_7$

TABULAR COLUMN:

Volume of $K_2Cr_2O_7$ (V)	E	ΔE	ΔV	ΔE/ΔV

Calculations:

$N_1V_1(FAS) = N_2V_2\ (K_2Cr_2O_7)$

$$N1 = \frac{N_2 \text{ X B.R (From graph)}}{25} = \frac{0.5 \text{ X B.R}}{25}.$$

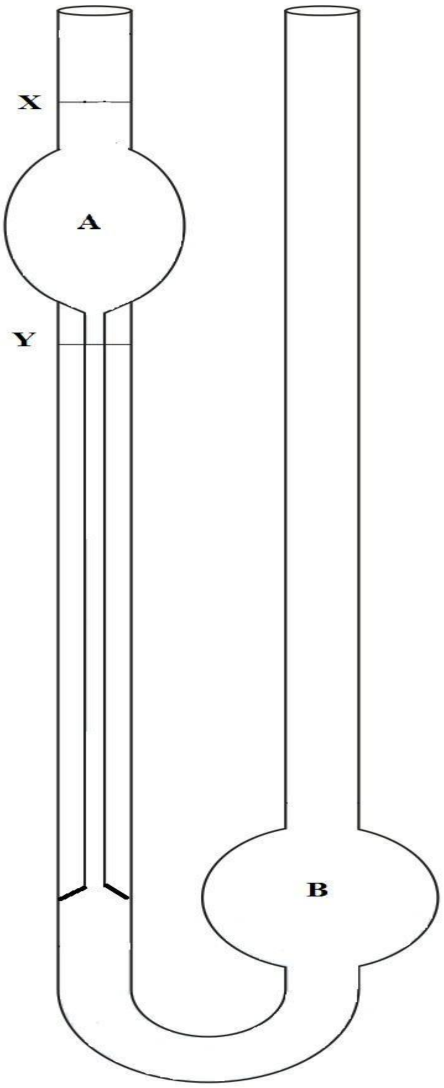
N_1=____________________N

Amount of FAS present in the solution = N_1X 392

=___________gm/dm^3

RESULT: Amount of FAS present in the given solution _________gm/dm^3

OSTWALD'S VISCOMETER



Viscometer

5. DETERMINATION OF VISCOSITYCOEFFICIENT OF CONDUCTIVE INKS (ANY LIQUID)

Experiment No: **Date:**

EXPERIMENT: Determination of viscosity coefficient of a given liquid using Ostwald's Viscometer.

REQUIREMENT: Ostwald's Viscometer, Stop watch, graduated pipette and hot-ait blower.

CHEMICALS: Reference liquid, Test liquid and acetone.

PRINCIPLE: Viscosity arises due to internal friction between moving layers of molecules. A liquid flowing through a cylindrical type of uniform diameter is expected to move in the form of molecular layers. A layer close to the surface is almost stationary while that at the axis of the tube moves faster than any other intermediate layer. As slow moving layer exerts friction on its nearest moving layer backwards. This property of a liquid by which it opposes motion between layers is called viscosity gradient between any two successive layers of a liquid situated at a unit distance apart. The coefficient of viscosity of liquid is given by poiseuille's formula.

$$V = \frac{\pi \rho r^4 t}{8 \eta l}$$

Where V=Volume of liquid, r =radius of the capillary, P is the pressure difference between the two ends of the tube, η is the coefficient of viscosity of the liquid. If equal volumes of two different liquids are allowed to flow through the same tube identical conditions then.

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

The time t_1 taken by the given liquid to travel through a certain distance in the tube is determined. The time t_2 taken by a reference liquid to travel through the same distance is measured. Knowing the densities d_1 and d_2 of the test liquid and the reference and also the coefficient of viscosity of the reference, coefficient of viscosity of the test liquid can be calculated.

PROCEDURE:

Clean the viscometer with distilled water acetone and dry it. Pipette out definite known volume (15 or 25ml depending upon the size of the bulb) of the reference liquid into the bulb "B". Suck the reference liquid into the upper bulb "A" until it rises above the mark "X". Now allow the reference liquid to flow freely through the capillary upto the mark "X". Start the stop watch and note the time "t_2" for the flow the liquid from mark "X" to mark "Y". Repeat for two more times.

Tabular Column:

Liquids	Time in Seconds				
		1	**2**	**3**	**Mean**
Test	**t_1**				
Reference	**t_2**				

Calculations:

$$\eta_1 = \eta_2 \times \frac{t_1 d_1}{t2d2}$$

Where:

η_1= Viscosity co-efficient of test liquid

η_2 =Viscosity co-efficient of reference liquid

d_1= Density of test liquid

d_2= Density of reference liquid

t_1= Time of flow of test liquid

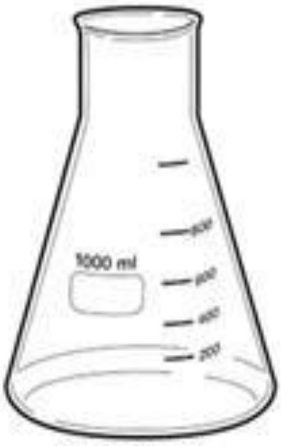
t_2= Time of flow of reference liquid

Remove the reference liquid from the viscometer, rinse with acetone and dry it. Now pipette out the same known volume of the test liquid as before into the bulb "B" of the viscometer and find out the flow of time "t_1" as before. Calculate the co-efficient of viscosity of the test liquid.

RESULT: Viscosity coefficient of test liquid =____________________millipoise**.**

PART-B

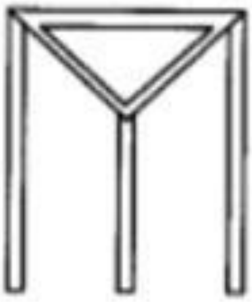
Volumetric Experiments



Conical Flask

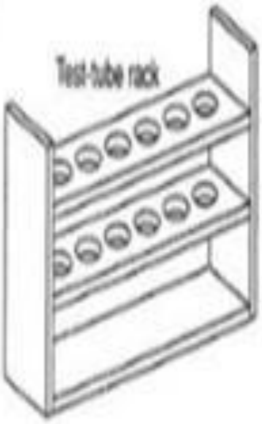
Beaker

Funnel



Tripod stand

Funnel stand

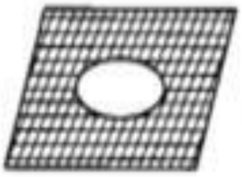


Buret

Volumetric pipet

Glass rod

Test tubes



Wire gauze

Watch glass

Test tube brush

Glass tube

Dropper

INSTRUCTION TO BE FOLLOWED FOR THE VOLUMETRIC ANALYSIS

BURETTE:

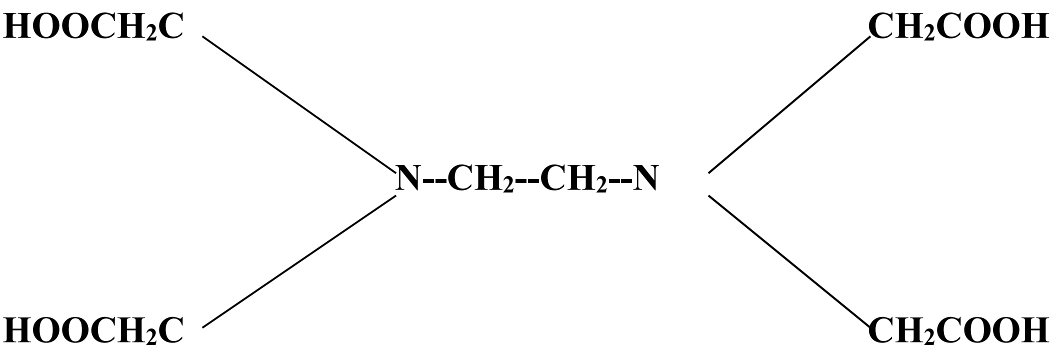
- Wash the burette with tap water and then rinse with distilled water.
- Rinse burette with a small quantity of the solution to be taken in burette and discard the solution into sink.
- Fill the burette with the solution using a small funnel which is already washed with tap water, rinsed with distilled water and then with the solution.
- Remove the funnel from the burette, before noting initial / final readings.
- Ensure that the nozzle of the burette is filled with the solution.
- Record the initial& final recordings properly.
- See that the level of the burette solution is at your eye level to avoid parallax error. (Similarly for pipette also).
- During the addition of the solution from the burette, the conical flask must be constantly swirled with one hand while the other hand controls the stop cock of the burette.

PIPETTE

- Wash pipette with tap water and then rinse with distilled water.
- Rinse pipette with a small quantity of solution to be taken in the pipette and discard the same into sink.
- During transferring solution into a clean conical flask, when all the solution from the pipette flows out, touch the tip of the pipette to the bottom of the flask gently.
- Do not blow out the last drops of the liquid from the pipette.

CONICAL FLASK

- Wash the conical flask with tap water and then rinse with distilled water.
- Do not try to drain out the water particles from the conical flask.
- DO NOT RINSE THE CONICAL FLASK WITH THE SOLUTION TO BE TAKEN IN THE CONICAL FLASK.
- During the titration, place the conical flask on a glazed white tile below the burette.



Structure of E D T A

OBSERVATION:

Weight of the substance = W =______________gm

$$\text{Molarity of EDTA} = \frac{\text{Weight of the substance}}{\text{Molecular weight of Na}_2\text{ EDTA}} \times 4$$

$$Z = \frac{W}{372} X\ 4 = \_\_\_\_\_\_\_\_\_\_\_ \text{M}$$

1. ESTIMATION OF TOTAL HARDNESS OF WATER BY EDTA METHOD

Experiment No: **Date:**

EXPERIMENT: Estimation of total hardness of water using EDTA by Complexometric method.

CHEMICALS: Na_2EDTA crystals, Buffer, Erichrome Black – T indicator.

PRINCIPLE: Hardness of water is due to the presence of calcium and magnesium salts in water. Ethylene diamine tetra acetic acid (EDTA) forms complexes with large number of cations including Ca^2 + Mg^2 + ions. Accordingly, it is possible to determine the total hardness of water using EDTA reagent.

The EDTA molecule (H_4Y) has two equally replaceable hydrogen atoms and the resulting ion after ionization may be represented as H_2Y^{-2} this forms complexes with metal ions.

$$M^{+2} + H_2Y^{-2} \longrightarrow MY^{-2} + 2H^+$$

Where M^{+2} is a Ca^{+2} and Mg^{+2} in water. Reaction can be carried out quantitatively at a pH of 10 using Erichrome black – T indicator, Since the reaction involves the liberation of H^+ ions, a buffer mixture has to be used to maintain a pH of 10. The buffer mixture used in the titration is NH_3-NH_4 Cl. The hardness of water is usually expressed in terms of ppm (parts per million) of $CaCO_3$ since EDTA (free acid) is sparingly soluble, its disodium salt, Na2 EDTA is used for preparing the agent.

PROCEDURE:

Part-I

Prepare standard solution of EDTA (Ethylene Diamine Tetra Acetic acid) by dissolving W gm of EDTA crystals in a 250 ml volumetric flask using distilled water

OBSERVATIONS:

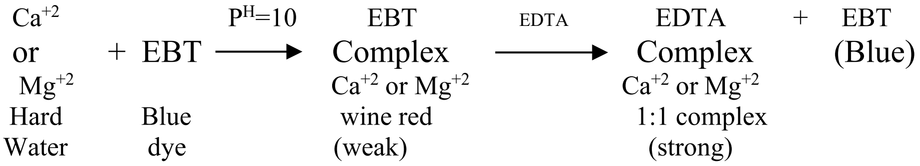
Burette : Standard EDTA solution

Conical : 25 ml of water sample + 2ml of buffer + 2-3 drops of EBT Indicator

Indicator : Erichrome Black - T

Colour change : Wine red to clear Blue

REACTION:



$$\underset{\substack{\text{Hard}\\ \text{Water}}}{\overset{Ca^{+2}}{\underset{Mg^{+2}}{\text{or}}}} + \underset{\substack{\text{Blue}\\ \text{dye}}}{\text{EBT}} \xrightarrow{P^{H}=10} \underset{\substack{Ca^{+2}\text{ or }Mg^{+2}\\ \text{wine red}\\ \text{(weak)}}}{\overset{\text{EBT}}{\text{Complex}}} \xrightarrow{\text{EDTA}} \underset{\substack{Ca^{+2}\text{ or }Mg^{+2}\\ \text{1:1 complex}\\ \text{(strong)}}}{\overset{\text{EDTA}}{\text{Complex}}} + \overset{\text{EBT}}{\text{(Blue)}}$$

TABULAR COLUMN:

Burette reading	I	II	III
Final			
Initial			
Difference in ml			

$$\text{Mean Burette Reading} = \frac{\text{I} + \text{II} + \text{III}}{3}$$

CALCULATIONS:

1 mole of EDTA = 1 mole of Ca^{+2} or Mg^{+2} salts

1 mole of EDTA = 1 mole of $CaCO_3$ equivalent

Total hardness = B.R x Z x 4000

=________________ppm

Part- II

ESTIMATION OF TOTAL HARDNESS:

Transfer 25 ml of the given water sample into clean conical flask using pipette. Add 2ml of buffer solution and 2-3 drops of EBT indicator, the contents of the flask are titrated against solution of EDTA till the wine red colour changes to clear blue.

RESULT: Total hardness of a given water sample =________________________ppm

Weight of the FAS: W =_______________g

Strength of FAS: $Z = \frac{W \times 4}{\text{Eq.wt of FAS}}$ (Eq. wt of FAS 392)

Z =________________N

OBSERVATIONS:

Burette : Standard solution of FAS

Conical Flask : 25 ml water sample + 25 ml $K_2Cr_2O_7$ solution 20 ml H_2SO_4

Indicator : Ferroin

Colour change : Green to reddish brown

TABULAR COLUMN:

Burette reading	I	II
Final		
Initial		
Difference in ml		

Mean Burette Reading = $\frac{I+II}{2}$ = ________________ml

Volume of FAS required for main titration = M =_______ml

2. DETERMINATION OF CHEMICAL OXYGEN DEMAND OF INDUSTRIALWASTE WATER SAMPLE.

Experiment No: **Date:**

EXPERIMENT: Determination of Chemical Oxygen demand (COD) of industrial waste water

CHEMICALS: $K_2Cr_2O_7$, H_2SO_4, FAS

THEORY: Chemical oxygen demand as the name implies it is the oxygen requirement of a sample for oxidation of organic and inorganic matter. As the oxidisable inorganic matter, is usually negligible in comparison with the quantity of organic matter, COD is generally considered as the oxygen equivalent of the amount of organic matter oxidisable by the potassium dichromate. This test is highly useful to find out the pollution strength of industrial effluent sewage.

The organic matter of the sample is oxidized with excess of potassium dichromate on a 50% H_2SO_4 (the excess $K_2Cr_2O_7$ is titrated with standard solution of ferrous ammonium sulphate solution) in the presence of Ag^+ ions catalyst and mercuric sulphate is added to eliminate the interference due to chlorides.

A known volume of the waste water sample is treated with excess of acidified $K_2Cr_2O_7$ unreacted $K_2Cr_2O_7$ is titrated with standard ferrous ammonium sulphate solution using ferroin indicator. A blank titration without the water sample is determined from the difference in the titrate values.

Volume required for blank titration B=_______________ml

CALCULATION:

Volume of FAS equivalent to the oxidisable impurities = (B-M) = V =____________ml

100 ml of 1N FAS solution = 1 equivalent of oxygen

100ml of 1N FAS solution = 8gms of oxygen

V ml of ZN FAS solution = 8 x V x Z mg of oxygen

I.e. 25 ml of water sample = 8 x V x Z x mg of oxygen

Therefore 1000 ml of waste water sample = 8 x V x Z x 40mg of oxygen

=__________mg of oxygen

PROCEDURE:

MAIN TITRAION

Weigh out accurately about 0.98 g of ferrous ammonium sulphate into a 250 ml volumetric flask. Add 1 test tube full of dilute sulphuric acid and shake the flask. Makeup to the mark with the distilled water mix the solution well. Calculate the normality of ferrous ammonium sulphate.

Pipette out 25ml of the waste water sample into the conical flask. Add 25ml of standard $K_2Cr_2O_7$solution (using burette). Add20 ml of 50% sulphuric acid (containing Ag_2SO_4 and $HgSO_4$) with constant shaking of the contents. Add few boiling chips to the flask. Boil the solution in the flask for about 15 minutes and cool it. Add 2-3 drops of ferroin indicator and titrate against FAS solution till the green colour changes to reddish brown.

BLANK TITRATION

Pipette out 25ml $K_2Cr_2O_7$ solution into a conical flask. Add 20ml of 50% sulphuric acid (containing Ag_2SO_4 and $HgSO_4$) Add 2-3 drops of ferroin indicator and titrate against standard ferrous ammonium sulphate solution till the solution turns from green colour to red colour.

RESULT: COD of the industrial waste water sample =______________mg of oxygen.

OBSERVATIONS:

Burette : Standard sulphuric acid

Conical Flask :25ml of water sample +2-3drops of phenopthalein indicator + 2-3 drops of methyl orange indicator

Indicator : phenopthalein & methyl orange indicator

End point : First step: Pink to colour less
Second step: re-appearrance of pink colour

TABULAR COLUMN :

Burette reading	**I**	**II**	**III**
Final			
Initial			
Difference in mL			

Mean burette reading = $\frac{\text{I+II+III}}{3} =$ _____________ml

CALCULATIONS:

Volume of water sample taken = 25 ml

Concentration of H_2SO_4 = 0.02N

Volume of consumed = V = ------------- ml

Total alkalinity of water sample = $\frac{\text{V} \times \text{Normality of } acid}{25}$

Total alkalinity of water sample in terms of $CaCO_3$ equivalent } = $\frac{\text{V} \times 0.02 \times 50}{25}$ g/l

= $\frac{\text{V} \times 0.02 \times 50 \times 1000}{25}$

= ____________________ mg/l

3. DETERMINATION OF TOTAL ALKALINITY OF GIVEN WATER SAMPLE

Experiment No: **Date:**

EXPERIMENT: Determination of total alkalinity of the given water sample

CHEMICALS: 0.02N H_2SO_4, Phenopthalein and Methyl Orange Indicator

PRINCIPLE: The alkalinity of water is due to the presence of hydroxyl ions (OH), carbonate ions $(CO_3)^2$ and bicarbonate ions $(HCO_3)^1$, the alkalinity of water can be estimated by titrating water with a standard acid using Phenopthalein and Methyl Orange Indicators reactions are as follows.

a) $OH^- + H \longrightarrow H_2O$
b) $CO_3^{2-} + H^+ \longrightarrow HCO_3^-$
c) $HCO_3^- + H^+ \longrightarrow H_2O + CO_2$

The titration of the water sample with a standard acid up to the Phenopthalein end point marks the completion of reaction a & b only, the amount of acid consumed corresponds to hydroxide plus one half of the carbonate ions present. Titration of the water sample with a standard acid up to the Methyl Orange end point marks the completion of all the three reactions i.e., (a, b % c). Hence the amount of the total acid consumed correspond to the total alkalinity.

PROCEDURE: 25 ml of the water sample into a clean conical flask, add 2-3 drops of phenopthalein indicator, titrate with standard sulphuric acid till the pink colour just disappears, then add 2-3 drops of methyl orange indicator to the same solution and titrate further with the standard acid till pink colour re-appears.Note down the burette reading.

RESULT: Total alkanity of the water sample = __________________ ml

OBSERVATION AND CALCULATION

PART-A:

Preparation of standard $K_2Cr_2O_7$ solution :

Weight of the Substance = W = _______g

$$N_{K_2Cr_2O_7} = \frac{W \times 10}{\text{Equivalent weight of } K_2Cr_2O_7}$$

$$= \frac{W x 10}{49} \quad (\text{ Equivalent weight of } K_2Cr_2O_7 = 49 \text{ g/l })$$

= __________ N

4. ESTIMATION OF IRON IN TMT BAR STEEL BY DIPHENYL AMINE INDICATOR METHOD

Experiment No: **Date:**

AIM: To determine the amount of iron present in TMT bar using standard solution of potassium dichromate.

CHEMICALS : TMT bar solution, potassium dichromate, sulphuric acid, phosphoric acid , HgCl2 , $SnCl_2$ and diphenylamine.

APPARATUS: Standard flask, burette, pipette, conical flask, test tube and beaker.

THEORY: Iron in TMT bar is treated with conc. HCl to convert metallic iron if any into chlorides.

$$2Fe + 6HCl \longrightarrow 2FeCl_3 + 3H_2\uparrow$$

Insoluble residues are removed by filtration. iron present in the solution as Fe^{3+} is reduced to Fe^{2+} using stannous chloride in hot condition in presence of concentrated HCl.

$$FeCl_3 + SnCl_2 \longrightarrow 2\,FeCl_2 + SnCl_4$$

The solution is cooled and excess stannous chloride is removed by adding saturated mercuric chloride.

$$SnCl_2 + 2\,HgCl_2 \longrightarrow Hg_2Cl_2\downarrow + SnCl_4$$

The Fe^{2+} ion in the resulting solution is titrated against standard $K_2Cr_2O_7$ solution using diphenyl amine as an indicator.If excess of stannous chloride is added, Hg2Cl2 will be reduced to Hg.

$$SnCl_2 + Hg_2Cl_2 \longrightarrow SnCl_4 + 2Hg$$

PROCEDURE:

PART A

Preparation of standard $K_2Cr_2O_7$ solution :

Weigh out given potassium dichromate crystals and transfer it to 100 ml standard flask. Dissolve it in distilled water, dilute up to the mark using distilled water and mix well. Calculate the normality.

=

PART-B:

ESTIMATION OF IRON IN TMT BAR :

Burette : $K_2Cr_2O_7$ solution

Conical flask :25ml of TMT bar sample solution+ 5ml conc. HCl, heat + $SnCl_2$, cool +3ml $HgCl_2$ +2ml of phosphoric acid

Indicator : Diphenylamine

Endpoint : Green to violet

TABULAR COLOUMN :

Burette reading	**I**	**II**	**III**
Final			
Initial			
Difference in mL			

Mean burette reading = $\frac{I+II+III}{3} =$ _____________ml

CALUCULATION:

1.Normality of TMT Bar Solution = $\frac{\text{B.R x Normality of } K_2Cr_2O_7}{25}$

= _____________________ N

2. Amount of iron in TMT Bar solution = Normality of TMT Bar Solution x Eq. Wt of iron

= Normality x 55.85

= ______________ x 55.85

= _______________ g/l

PART-B

Estimation of Iron:

Pipette out 25 ml of the TMT bar solution into a clean conical flask. Add 3ml of concentrated HCl and heat nearly to boil. Add $SnC1_2$ drop wise to the hot solution from the burette till the solution becomes colorless. Add 2drops in excess and cool the solution under the tap water. Add 3ml of saturated $HgCl_2$ rapidly in one portion. A silky white precipitate should be formed (if black precipitate is formed or if no precipitate is formed, discard the solution and repeat the experiment). Add 2ml of phosphoric acid. Titrate against standard $K_2Cr_2O_7$ solution using diphenylamine as an indicator . The colour turns green to violet at the end point. Repeat the titration for concordant values and calculate the percentage of iron.

RESULT: Amount of iron in TMT bar solution =________________ g/l

OBSERVATIONS AND CALCULATIONS:

Weight of bottle and sodium oxalate crystals: --------(W1) g

Weight of empty bottle: ----------------(W2)g

Weight of Sodium Oxalate Crystals= ---------- (W2-W1) g

$$N = \frac{(W2-W1)\ X\ 4}{Eq\ Wt\ of\ Sodium\ Oxalate} = \frac{\quad X\ \ 4}{67}$$

N = ____________ N

PART A

TABULAR COLUMN :

Burette reading	**I**	**II**	**III**
Final			
Initial			
Difference in mL			

Mean burette reading $V_1 = \frac{I+II+III}{3} =$ _____________ml

$(N_1V_1)\ KMnO_4 = (N_2V_2)\ Na_2C_2O_4$

$$N_{KMnO4} = \frac{N(Na2C2O4\ )\ X\ 25}{BR}$$

$N_{KMnO4} =$ ____________N

5A . DETERMINATION OF HYDROGEN PEROXIDE: TITRATION BASED ON OXIDATION AND REDUCTION

Experiment No: **Date:**

AIM: Determination of Hydrogen Peroxide using Standard Potassium Permanganate

PRINCIPLE: Hydrogen peroxide reacts with permanganate ion in acidic solution according to

$$5\ H_2O_2 + 2\ MnO_4^- + 6\ H^+ \rightarrow 2\ Mn^{2+} + 5O_2 + 8\ H_2O$$

Hydrogen peroxide in a diluted portion of the sample is quantitatively oxidized by titration with a potassium permanganate solution of known strength. Compounds that are oxidized by potassium permanganate under acidic conditions interfere.

PROCEDURE:

PART A

Standardization of permanganate solution against sodium oxalate Anhydrous sodium oxalate
Preparation of 0.05 M sodium oxalate :

Weigh 1.7 g of sodium oxalate, transfer into a 250 mL volumetric flask, add about 200 mL of DW, swirl until complete dissolution, and dilute to mark and mix well and calculate the molarity of the solution.

Standardization of permanganate:

Pipette 25.00 mL of sodium oxalate standard solution into a conical flask, and add 20 mL of 1:10 sulfuric acid and then 75 mL of water, heat the solution slowly on a hot plate, with stirring, to about 80 °C (use a thermometer to determine temperature, but not as stirring rod!). The hot solution was titrated against 0.02M Potassium permanganate solution until a faint pink colour persists. Repeat for concordant ridings.

Part B

Tabular Column :

Burette reading	**I**	**II**	**III**
Final			
Initial			
Difference in mL			

Mean burette reading V = $\frac{I+II+III}{3}$ = ____________ml

$(N_1V_1)_{H2O2} = (N_2V_2)_{KMnO4}$

$N_{H2O2} = \frac{N(KMnO4)\ X\ V}{25}$

N_{H2O2}= _________N

Weight of H_2O_2= N_{H2O2} X Eq.Wt of H_2O_2

= ______ X 17

= __________g/l

Determination of hydrogen peroxide:

Transfer the sample to a 500-mL volumetric flask containing about 250 mL of water and 2 mL of H2SO4 (1:3). Thoroughly rinse the beaker into the volumetric flask. Dilute to volume with water and mix well. Pipette 25.0 mL of the solution into a 250-mLflask containing 10 mL of H2SO4 (1:3) and 60 mL of water. Add the standardized potassium permanganate solution from a 50-mL Class-A burette until the first appearance of a faint pink colour that persists for 30 seconds. Record the volume delivered as **V**.

RESULT: Amount of H_2O_2 present in a given sample = ____________g

CALCULATION:

The acid number was calculated as follows:

$$\text{acid value}\left(\frac{\text{mg KOH}}{\text{g sample}}\right)=\left[\frac{\text{volume KOH(mL)}\times\text{N KOH(mmol/mL)}*56.1\text{(mg/mmol)}}{\text{sample weight (g)}}\right]$$

Weight of the biodiesel sample = __________ g

N of KOH used = 0.02 M Vol of KOH

consumed in titration = ______ ml

5B. DETERMINATION OF ACID VALUE OF BIOFUEL

Experiment No: **Date:**

EXPERIMENT: Determination of acid value of biodiesel

CHEMICALS: The soybean oil, IPA, toluene, sulfuric acid, KOH, indicator (Phenolphthalein)

THEORY: This test method covers procedures for the determination of acidic constituents in petroleum products and lubricants that are soluble in mixtures of toluene and isopropanol. In B100 (biodiesel), the acid number is a measure of free fatty acids. The free fatty acids can lead to corrosion and are a symptom of water in the fuel or fuel oxidation. The acid value is defined as the number of milligrams of Potassium hydroxide required to neutralize the free fatty acids present in one gram of fat. It is a relative measure of rancidity as free fatty acids are normally formed during decomposition of triglycerides. The value is also expressed as per cent of free fatty acids calculated as oleic acid, lauric, ricinoleic and palmitic acids.

PRINCIPLE: The acid value is determined by direct titration of oil/fat in an alcoholic medium against standard potassium hydroxide/sodium hydroxide solution.

PROCEDURE: For determining the acid number, take 2 g of a biodiesel sample in an Erlenmeyer flask (125 mL), then add 10ml of titration solvent and 0.5 mL of the indicator solution and mix thoroughly. The sample was then titrated against 0.02N KOH solution. The titration was deemed complete when a color change from orange to green that held for at least 15 s was observed in the titration mixture

RESULT: The acid number of the biodiesel sample = ___________

OBSERVATION AND CALCULATIONS:

Preparation of Standard EDTA

1. Weight of the bottle + EDTA crystal W1=..................... g
2. Weight of the bottle after transfer W2 = ... g
3. Weight of EDTA crystals taken (W1-W2) = W= .... g

$$M_{EDTA} = \frac{W \times 4}{\text{Molecular weight of EDTA}} = \frac{W \times 4}{372} = \frac{\text{----------} \quad \times 4}{372}$$

$$= \underline{\qquad\qquad} \ M\ (A)$$

5C. ESTIMATION OF PERCENTAGE OF CaO IN CEMENT BY COMPLEXOMETRIC METHOD

Experiment No: **Date:**

AIM: To estimation the percentage of CaO in Portland cement.

THEORY: Cement contains oxides of Calcium, magnesium, silica and small quantities of aluminum &iron. Patton and Reeder's indicator permits the determination of calcium in the presence of the magnesium at pH-12. The interference of magnesium ions is avoided by precipitating them as hydroxides by the addition of 4 N NaOH. The pH of the solution is maintained at 12-14 by adding diethylamine. A sharp end point obtained by adding 1:1 glycerol. The colour changes from wine red to clear blue when calcium ions are titrated with EDTA solution.

PROCEDURE:

PART A

Preparation of standard solution of di-sodiumsalt of EDTA :

Weigh the given di-sodium salt of EDTA crystals into 250 ml standard flask, dissolve the salt using 10 ml ammonium hydroxide. Dilute up to the mark using distilled water, mix well and calculate the molarity.

ESTIMATION OF CALCIUM OXIDE

Burette : EDTA Solution

Conical flask : 25ml of cement solution + 2ml of1:1glycero l+ 2ml of diethylamine + 5 ml of 4 N NaOH

Indicator : Patton and Reeder's

End point : Wine red to blue

TABULAR COLOUMN:

Burette reading	I	II	III
Final			
Initial			
Difference in mL			

Volume of EDTA consumed = $\frac{I+II+III}{3}$ = _________(B) ml

Wight of CaO per liter = 'c' g= _____________ g (to be given)

M cement solution = $\frac{(M \times V)EDTA}{V_{\text{cement solution}}} = \frac{A\times B}{25}$ = __________________

= _______________ M (D)

Wight of CaO per liter of cement solution = $M_{\text{cement solution}} \times$ molecular weight of CaO

= D×56.08

= ___________×56.08

= ____________g (E)

Percentage of CaO in cement = $\frac{\text{Weight of CaO per liter}}{\text{Weight of cement per liter}} \times 100 = \frac{E\times B}{C}$

=___________×100

=_____________%

PART B

Estimation of Calcium Oxide:

Pipette out 25 ml of the given cement solution in to a clean conical flask. Add 2 ml of 1:1 glycerol with constant shaking followed by 2ml of diethylamine (to adjust the pH of the solution to 12). Add 5ml of 4N NaOH to precipitate magnesium as Mg(OH)2. Mix the solution well. Add 2drops of Patton and Reeder's indicator. Titrate against EDTA solution till the colour changes from winered to clear blue.(Perform the titration very slowly near the end point). Let the volume consumed be 'B' ml. Repeat the titration for concordant values.

RESULT: The percentage of CaO in the Portland cement =________%