SRI INDU INSTITUTE OF ENGINEERING AND TECHNOLOGY

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KhalsaIbrahimpatnam, Sheriguda(V), Ibrahimpatnam(M), Ranga Reddy Dist., Telangana – 501 510

DEPARTMENT OF HUMANITIES AND SCIENCES

ENGINEERING CHEMISTRY LAB   MANUAL

ENGINEERING CHEMISTRY LAB (BR22)

<u>CERTIFICATE</u>

This is to Certify That **Prepared by Dr.D.Premalatha, G.Vijay, K.Mounika,O.Subhashini, V.Mounika,** *have prepared manual of practical work done in the Engineering Chemistry laboratory ,.*

Lab Incharge   H.O.D

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B.Tech. I Year I Sem   **L T P   C**

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VIII. 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.

REFERENCE BOOKS:

1. Lab manual for Engineering chemistry by B. Ramadevi and P. Aparna, S Chand Publications, New Delhi (2022)

 2. Vogel's text book of practical organic chemistry 5th edition

 3. Inorganic Quantitative analysis by A.I. Vogel, ELBS Publications.

 4. College Practical Chemistry by V.K. Ahluwalia , Narosa Publications Ltd. New Delhi (2007).

Instructions for Record Writing

1. Write on the right hand page the following in order:

   a) Serial number and date of performance (in the margin)

   b) Name and number of the experiment as given in the list

   c) Aim of the experiment

   d) Description of the apparatus

   e) Procedure including sources of error and precautions taken to eliminate or to minimize them.

   f) Inference or Result.

   g) Explanation, if necessary of any divergence in the expected result.

2. Left hand page should contain the following in their proper places.

   a) Neat diagram of the main apparatus used

   b) Observation in tabular form

   c) Calculation in tabular form

   d) Graph sheets and other papers to be attached.

N.B. (a) Students should submit a record of the previous experiments when they come for practical work.

   (b) An experiment is deemed to be complete when it is satisfactorily performed and recorded.

   (c) All suggestions given or defects noted should be attended to when the records are taken back.

KEEP THE RECORD BOOK NEAT IT FETCHES MARKS

EXPERIMENTS

Determination of Total Hardness of Water by Complexometric Method Using EDTA

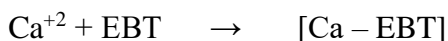**Aim :** To estimate the total hardness of water by EDTA Method.

Outcome : The students will be able to analyse the nature of salts causing hardness and to solve the engineering problems arising during steam production in Boilers.

Chemicals Required: Ammonia buffer solution, Eriochrome Black – T (EBT), Eriochrome Black – T (EBT), Magnesium chloride, EDTA Solution, Hard water

**Apparatus:** Conical Flask, Pipette, Burette, Beaker

Principle:
Ethylene diammine tetra acetic acid (EDTA) forms stable complexes with Ca^{+2} and Mg^{+2} ions present in water at pH 9-10. The sample of hard water must be treated with ammoncal buffer solution and EBT – indicator which forms unstable, wine red coloured complex with Ca^{+2} and Mg^{+2} present in water.

$$Ca^{+2} + EBT \quad \rightarrow \quad [Ca – EBT]$$

(Blue Colour)   (Complex, wine red colour)

$$Mg^{+2} + EBT \quad \rightarrow \quad [Mg – EBT]$$
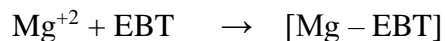
(Blue Colour)   (Complex, wine red colour)

The stability of a metal indicator complex is less than that of metal EDTA complex. During the titration of the complex with EDTA, EDTA extracts the metal ions from the metal – ion – EBT indicator complex and forms stable, colorless complexes by releasing the free indicator. Where by the end point of the titration is the colour change from wine red to blue.

$[(Ca^{+2} – EBT) + (Mg – EBT)] + EDTA \quad \rightarrow \quad (Ca – EDTA) + (Mg – EDTA)] \quad + \quad EBT$
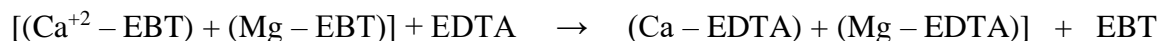
Wine red coloured complex   Colorless & stable complex   Blue in Colour

$[MEDTA]^{+n}$
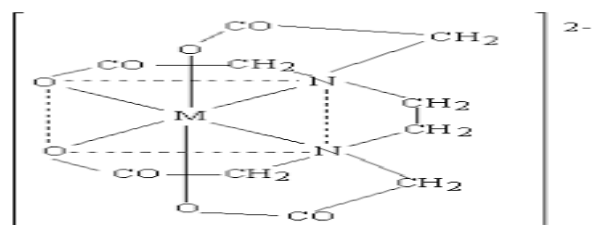
Procedure:

Preparation of standard hard water*:*

Dissolve one gram of pure, dry $CaCO_3$ in minimum quantity of diluted HCl and evaporate the solution to dryness on water bath. Dissolve the residue in small amount of water and transfer into a 100ml standard flask. Makeup the solution to the mark with distilled water and shake the flask well for uniform concentration.

Standardization of EDTA Solution:

Pipette out 10ml of standard hard water solution in to a conical flask, add 2 ml of buffer solution and 2-3 drops of EBT indicator and titrate the wine red coloured complex with EDTA solution taken in the burette, after rinsing the burette with EDTA solution, till the wine red colour of the solution changes to blue colour. Note the burette reading repeat the titration to get concurrent values.

Estimation of hardness of sample water:

Pipette out 2 ml of the water sample in to a 25ml conical flask, add 2ml of buffer solution and 3 drops of EBT – indicator. Titrate the wine red coloured solution with EDTA taken in the burette, till a clear blue coloured end point is obtained. Repeat the titration to get concurrent values.

Observation & Calculations

Standardization of EDTA:

S.No.	Volume of hard water	Burette reading		Volume of EDTA
		Initial	Final	
1				
2				
3				

Estimation of Hardness:

S.No.	Volume of Sample hard water	Burette reading		Volume of EDTA
		Initial	Final	
1				
2				
3				

Molority of standard Hard water $CaCO_3$

$$M_1 = \frac{Wt\ of\ CaCO_3}{M.wt\ of\ CaCO_3} \times \frac{1}{Volume\ of\ solution}$$

$$= \underline{\hspace{1.5cm}} M$$

Molarity of EDTA Solution (M_2) =?

$$\frac{V1M1}{n1} = \frac{V2M2}{n2}$$

$$n_1 = n_2 = 1$$

$$M_2 = \frac{V1M1}{V2} = \underline{\hspace{2.5cm}} M$$

V_1 = Volume of standard hard water = _____ ml

V_1 = Volume of EDTA = _____ ml

M_1 = Molarity of standard hard water = _____ M

M_2 = Molarity of EDTA = _____ M

Molarity of the sample water = _____(M_3) or (M sample)

M_2V_2 = M_3V_3

$$M_3 = \frac{M2V2}{V3} = \underline{\hspace{1.5cm}} M$$

V_2 = volume of EDTA

M_2 = Molarity of EDTA

V_3 = volume of hard water

Total hardness of water = M_3 x 100 x 1000

$$= \underline{\hspace{1cm}} ppm.$$

Result:

The total hardness of sample of water = _____ ppm.

Estimation of the concentration of an acid by Conductometric Titration

Aim : To determine the strength of the strong acid by titration with strong base Conductometrically.

Outcome : The student shall be able to analyze the variation of conductance values of given acid with addition of strong base using conductivity meter .

Chemicals Required: Sodium hydroxide, Hydrochloric acid

Apparatus: Conductivity Bridge, Conductivity cell, Burette, Beakers, Standard flask, pipette, Burette Stand.

Principle:

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

$$H^+ + Cl^- + Na^+ + OH^- \longrightarrow Na^+ + Cl^- + H_2O$$
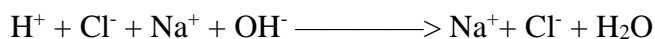
As NaOH is added the H^+ ions are removed. The conductivity decreases as Na^+ ions do not process much mobility. As the neutralization point and solutions contains Na^+ ions and Cl⁻ ions and will have minimum conductance value. If NaOH is further added this will add OH⁻ ions and so the conductivity increases.

Procedure:

A standard solution of 1M NaOH is prepared. Similarly, 0.1M HCl is prepared. 200 ml of HCl is taken in a 250 ml beaker. Further, the conductivity cell is washed with distilled water and rinsed with acid soln. The cell is kept in acid containing beaker and it is connected to the bridge. The conductivity of the solution is measured by adjusting the reading. NaOH solution is taken into burette and add 1 ml of solution to acid, stirred well and conductance is measured. Each time 1 ml of base is added to acid stirred well and the conductance is measured. For every instance equal numbers of values are taken on either side of the point of maximum. Repeat the procedure of addition of 1 ml NaOH and noting the conductivity of the resulting solution. Take 20-25 readings
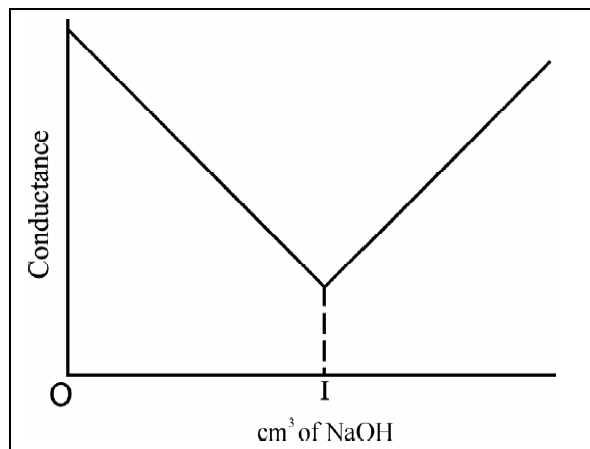
Calculations:

Volume of unknown acid (V_1) =200 ml.

S. No	Volume of NaOH (mL)	Observed conductance
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		

FORMULA:

$$N_1V_1 \text{ (HCl)} = N_2V_2 \text{ (NaOH)}$$

$$N_1 = \frac{1 \, X \, V_2}{200}$$

Strength of solution = Eq. Weight X Normality

$$= 36.5 \, X \, N_1$$

$$= \text{--------------- g/lit}$$

RESULT:

The Normality of strong acid (HCl) determined by titrating against a strong base (NaOH) =_____N

Precautions:

1. The conductivity cell should be handled very carefully as it is very delicate.

2. Stirring should be done after each addition of the titrant.

Advantages or applications of conductance titration:

1. This method can be used to very dilute solutions.

2. Gives very accurate end points with an error of $\pm\ 0.5$

3. These titrations are very useful in case of coloured solutions which cannot be titrated by ordinary volumetric method because colour change of indicator is not clear.

4. Useful of titrating weak acids against weak bases, which otherwise do not give sharp end points.

5. No keen observation is necessary near the end point since it is detected graphically.

EXPERIMENT-3

Estimation of the amount of Fe^{2+} by Potentiometry

Aim : To estimate the Fe^{2+} by Potentiometry using $KMnO_4$.

Outcome : The student shall be able to analyze the variation of EMF values of given acid with addition of $KMnO_4$ using Potentiometer.

Chemicals: $KMnO_4$, Distilled water, sulphuric acid, Ferrous sulphate solution.

Apparatus: volumetric flask, pt electrode, saturated calomel electrode, potentiometer

Principle:

Potentiometric titration is the titration in which potentiometric measurements are carried out in order to fix the end point. In this method, the interest is with the change in electrode potential, rather than with an accurate value for the electrode potential in a given solution. In a potentiometric titration, the change in cell e.m.f. occurs most rapidly in the neighbourhood of the end point.

The Fe(II) –$KMnO_4$ redox system is represented as

$5Fe^{2+} + MnO_4^- + 8H^+ \rightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$
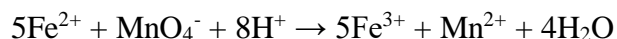
The determining factor is the ratio of the concentrations of the oxidised and the reduced forms of the iron species.

For the reaction,

Oxidised form + ne- $\rightarrow$ Reduced form,

$$E = E^o + \frac{0.0591}{n} \log \frac{[Ox]}{[Red]}$$

where E^0 is the standard Reduction Potential of the system. Thus the potential of the immersed electrode is controlled by the ration of these concentrations. During redox reactions, the potential changes more rapidly at the vicinity of the end point. The indicator electrode is usually a bright platinum wire or foil, the oxidising agent is taken in the burette. The cell can be represented as,

$$\text{Calomel} \| Fe^{3+}, Fe^{2+} | Pt.$$
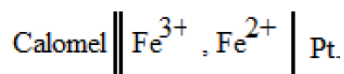
Here Pt is the indicator electrode and calomel is the reference electrode.

Procedure:

Preparation of 0.1 N $KMnO_4$:

0.1 N KMnO$_4$ is prepared by dissolving 0.31 g of analar crystals in distilled water in a 100 ml volumetric flask. The solution is made up to the mark.

Calibration of The Potentiometer:

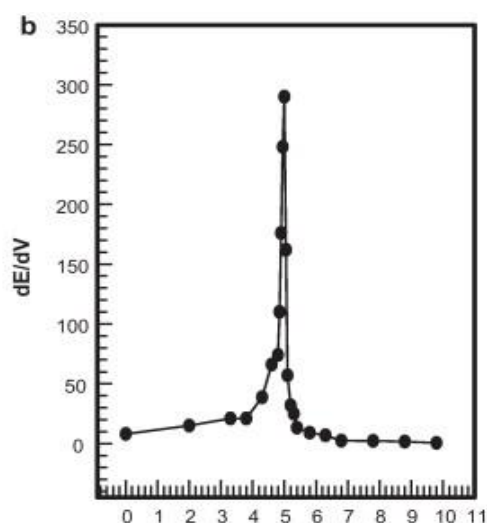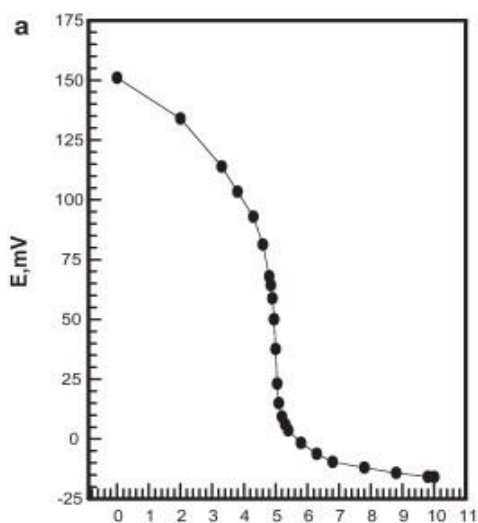A standard cell of known emf is connected to the instrument and its emf is set in the voltage scale. The galvanometer key is pressed to complete the circuit and the deflection of the galvanometer needle is noted. If there is any deflection, the current passing through the rheostat is adjusted for null deflection. This procedure makes sure that the value of emf which is read on the scale is the true potential of the cell considered. The potentiometer is calibrated using the Weston standard cell of potential 1.018 V.

Estimation Of Fe (Ii):

- The given Fe (II) solution is made upto 100 ml in volumetric flask.
- 20 ml of the solution is pipetted out into a clean beaker. To this, 25 ml of 2.5 M H$_2$SO$_4$ and 50 ml of distilled water are added.
- A platinum electrode is dipped into this solution, and it is coupled with a calomel electrode through a salt bridge. The resulting cell is connected to the potentiometer.
- Standard KMnO$_4$ solution is added from the burette, to this solution, insteps of 1 ml and the emf is recorded after each addition.
- At the end point, there is a jump in emf due to the absence of Fe^{2+}. The approximate range of the end point is determined.
- The experiment is repeated by adding the titrant in steps of 0.1 ml near the end point. A graph is plotted between emf, E and the volume of dichromate added. The inflexion point gives the volume of titrant at the end point.
- The first derivative ($\Delta E/\Delta V$ vs. Volume of titrant) curves give the exact volume of dichromate required for the reaction. From the plot of E vs. Volume of titrant, potential at the equivalent point is obtained.
- Atomic weight of Fe is 55.85

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Volume of KMnO$_4$

Table:

S.No	Volume of KMnO$_4$	EMF	ΔE	$\Delta E/\Delta V$	V $_{mean}$
1					
2					
3					
-					
-					

Volume of KMnO$_4$ = V$_1$

Concentration of KMnO$_4$ = N$_1$ = 0.1 N

Volume of Fe^{2+} solution = V$_2$ = 20 ml

Concentration of Fe^{2+} solution = N$_2$ = ?

According volumetric law N$_1$V$_1$ = N$_2$V$_2$

$$N_2 = N_1V_1/V_2$$

Amount of Fe^{2+} ion present in the given solution= (N$_2$ x Equivalent weight of Fe^{2+}) /10

$$= (N_2 \text{ x } 55.85/10) \text{ gm}$$

Result: The amount of Fe^{2+} ion present in the given solution=………..gm

EXPERIMENT-4

## Determination of an acid concentration using pH meter.

Aim: To estimate the amount of HCl present in the given volume of test solution by pH metry

Apparatus: Pipette, 50 mL Burette, 100 mL Beaker, pH meter, glass electrode

Chemicals: HCl, NaOH

Principle: pH of the solution is related to the H ion concentration of the solution by the expression

$+ \log [H- =pH] +$

The concentration of H ions in the solution is determined by measuring the pH of the solution. +When NaOH is added slowly from the burette to the solution of HCl, H ions are neutralized by ions. As a result, pH of the solution increases .

$$HCL+NaOH \rightarrow NaCl + H2O$$
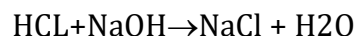
The increase in pH takes place until all the H. ions are completely neutralized. After the end point, .–further addition of NaOH increases the pH sharply as there is an excess of OH-ions

 Model Graph

 Procedure:

 1. Take 20 mL of acidified ferrous solution in a 100 mL beaker.

 2. The glass electrode is dipped into the solution and connected to the pH meter The ends of the electrodes are connected to the potentiometer.

 3. Note down the pH value of the solution.

4. Add 0.5 mL of NaOH taken in the burette to the HCl solution in the beaker.

5. After the addition of each 0.5 mL of K2Cr2O7, the solution is mixed well with a glass rod and note down pH value.

 6. After the sharp increase in pH value, take six readings by adding 0.5 mL of NaOH and note down pH values.

Observations:

S.NO	Volume of NaOH (mL)	PH	Δ pH	ΔV in mL	ΔpH/ΔV

Calculations:

A graph is plotted between pH and the volume of NaOH and also a first derivative graph is plotted (ΔpH/ΔV against vol. of NaOH). The strength of HCl solution and the amount of ferrous ion present are calculated from the end point.

N1V1 = N2V2

Volume of HCl (V1) = ...Ml

Normality of HCl (N1) =.......N

Volume of NaOH (V2) =........ mL

Normality of NaOH (N2) = ------N

Normality of HCl (N1) = (N2 x V2) / V1 =..........N

Amount of HCl present in1000 mL of the given solution = Strength (N1) x equivalent weight of HCl (36.5) = gm/lit

Result

Normality of HCl = ...N

The amount of HCl present in one litre of the solution is------------ gm/lit

EXPERIMENT-5

Preparation of Bakelite.

Aim: To prepare Bakelite polymer using Phenol and Formaldehyde

Apparatus: Beaker, glass rod, funnel, filter paper, and chemical balance.

Chemicals: Phenol, formaldehyde, conc. HCl, glacial acetic acid, distilled water.

Theory: Phenol formaldehyde resin is prepared by condensation polymerization of phenol and formaldehyde in the presence of an acidic or alkaline catalyst. They were first prepared by Backeland, an American Chemist who gave them the name as Bakelite. These are thermosetting polymers. The initial reaction involves formation of o- and p-hydroxy methyl phenol, which reacts to form linear polymer Novalac. The Novalac reacts formaldehyde to form hard infusible and insoluble solid of cross – linked structure known as Bakelite.

Synthesis of Bakelite
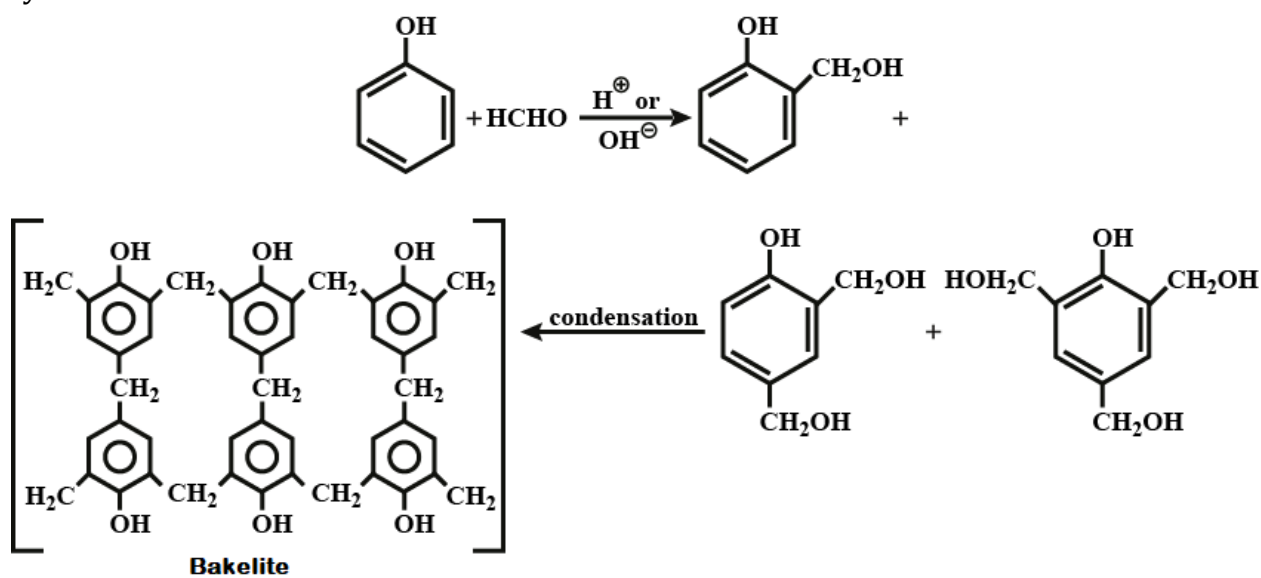
Procedure:

1. Place 5 mL of glacial acetic acid and 2.5 mL of 40% formaldehyde solution in 100 mL beaker.

2. Add 2 g of phenol to it.

3. Wrap a cloth loosely round the beaker. Add a few mL of conc. HCl in to the mixture carefully and heat itslightly.

4. A large mass of plastic pink in colour is formed.

5. A residue is washed with water and filtered.

6. The product dried and yield is weighed.

Precautions:

1. While adding conc. HCl, it is better to stay little away from the beaker since the reaction sometimes becomes vigorous.

2. The reaction mixture should be stirred continuously.

Observations:

Mass of the beaker, W1 = …………g

Mass of the beaker and phenol-formaldehyde resin, W2 = ………..g

Mass of phenol-formaldehyde resin = W2-W1= ………….g

Result:

The yield of phenol-formaldehyde (Bakelite) = --------g

EXPERIMENT-6

Synthesis of Nylon-6

Aim: To prepare Nylon-6, polymer using adipoyl chloride and hexamethylenediamine

Apparatus: Beaker, Glass stir rod

Chemicals: Hexamethylenediamine, Hexane, Adipoyl chloride, NaOH

Principle: Nylon 6,6 is prepared by condensation polymerization of adipic acid (or adipoyl chloride) and hexamethylenediamine. The 6,6 numbering refers to the number of carbon atoms in the adipic acid (or adipoyl chloride) and hexamethylenediamine respectively. In the experiment, there is a solution of adipoyl chloride in cyclohexane onto an aqueous basic solution of 1,6-hexanediamine (hexamethylenediamine). Because cyclohexane and water are immiscible, a two-layer system will result. The polymerization will take place at the cyclohexane-water interface. Sodium hydroxide is added to the reaction mixture to aid in the amide formation.

Synthesis of Nylon 6
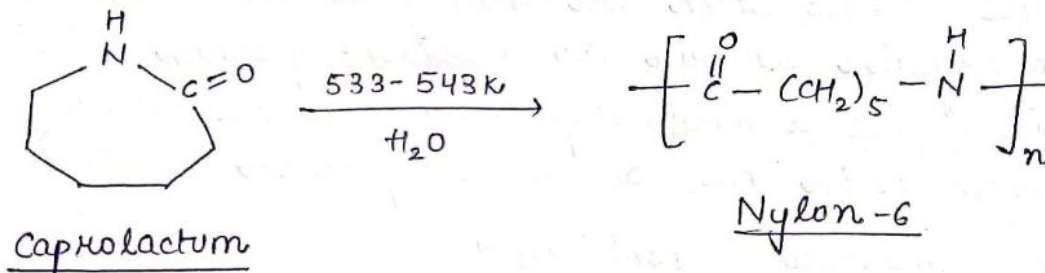
Procedure:

1. Place 10 mL of a 5% solution of adipoyl chloride in cyclohexane in a 50-mL beaker.

2. Place 10 mL of a 5% aqueous solution of hexamethylenediamine in a separate 150-mL beaker.

3. Add 7 drops of 20% sodium hydroxide solution to the hexamethylenediamine solution.

4. Slightly tilt the beaker containing the aqueous diamine solution and slowly pour the adipoyl chloride solution down the wall of the beaker to form two layers. Do not mix or stir. A film will form at the interface between the two solutions.

5. With forceps, grab a hold of the film at the center, pull slowly, and wind the resultant fiber onto a glass rod.

6. Rinse the nylon rope with water and dry the nylon fiber by sandwiching between paper towels

Result:

The yield of Nylon-6 = --------g

EXPERIMENT-7

Estimation of Acid Value of given lubricant oil

Aim : To Determine the Acid Value of Coconut Oil.

Outcome : The student shall be able to identify and calculate the acid values of coconut oil.

Chemicals: NaOH, distilled water, Phenolphthalein indicator, potassium hydrogen phthalate, coconut oil, ethanol, diethyl ether

Apparatus: Volumetric flask, conical flask pipette**,** burette, weighing balance, beaker

Principle:

Acidity may occur in coconut oil upon storage due to the decomposition of the oil to free fatty acids, peroxides and low molecular weight aldehydes and ketones, which produce distinctive smell and affect the quality of oil. Acid value may be defined as the number of milligrams of KOH required to neutralise the free acid in 1gm of fatty oil.

Procedure:

Preparation of Solutions:

1) **0.1 N Sodium hydroxide:** Weigh accurately 0.4gm of sodium hydroxide into 100mL standard flask, dissolve in little distilled water and make up the solution to the mark with distilled water and shake the flask well for uniform concentration.
2) **Ethanol-ether solution:** Prepare a mixture of ethanol and diethyl ether (1:1 v/v); add 1mL of phenolphthalein indicator and titrate with NaOH till pale pink colour is observed. This is the neutralisation of ethanol-ether solution.
3) **Standard solution of potassium hydrogen phthalate:** Accurately weigh about 1.2gm of potassium hydrogen phthalate into 100mL flask dissolve in little water make up the solution to the mark with distilled water.

Procedure:

1) **Standardisation of NaOH solution:** Pipette out 20mL of potassium hydrogen phthalate solution into a 250mL conical flask and add 2 drops of phenolphthalein indicator and titrate the solution with NaOH taken in burette. Pale pink colour is the end point of the titration. Note the burette reading and let the value be x mL
2) **Standardisation of coconut oil:** Accurately weigh 1gm of coconut oil into a 250mL conical flask; add 50mL of ethanol-ether solution. Shake the flask well for uniform dissolution of the sample. Add 1 drop of phenolphthalein indicator and titrate the solution with NaOH taken in the burette till pale pink colour is observed. Note the burette reading, let the value be y mL

Calculations:

(M_1) Molarity of potassium hydrogen phthalate $= \dfrac{1.2 \text{ X } 1000}{204.22 \text{ X } 100} = 0.058M$

 (M_2) Molarity of NaOH: M_1V_1/M_2V_2

$$M_2 = M_1V_1/x$$

Acid value of coconut oil $= \dfrac{M_{NaOH}}{W}$ (W = wight of coconut oil = 1 gm)

Result: The acid value of given oil is …………..

EXPERIMENT-8

Estimation of Viscosity of Lubricant Oil Using Ostwald's   Viscometer

Aim : To determine the viscosity of the given castor oil and ground nut oil.

Outcome : The student shall be able to determine and calculate the viscosity coefficient values of Castor oil and groundnut oil.

Chemicals: astor oil and groundnut oil, distilled water.

Apparatus: Specific gravity bottle, viscometer, rubber tube with screw pinch cock, stand, beaker

Theory: The force of friction which one part of the liquid offers to another part of the liquid is called viscosity. For measuring the viscosity coefficient, Ostwald viscometer method is used which is based on Poiseuille's law. According to this law, the rate of flow of liquid through a capillary tube having viscosity coefficient 'η' can be expressed as

$$\eta = \frac{\pi : r^4 tP}{8vl}$$

where, v= vol. of liquid (in ml)

t= flow time (in sec.) through capillary

r= radius of the capillary (in cm)

l= length of the capillary (in cm)

P= hydrostatic pressure (in dyne/sq.cm)

η= viscosity coefficient (in poise).

Since, the hydrostatic pressure (the driving force) of the liquid is given by P = dg h (where h is the height of the column and d is the density of the liquid);

$\quad$ η α P t ; or, η α dght

If, η_1 and η_2 are the viscosity coefficients of the liquids under study, d_1, d_2 are their densities and t_1 and t_2 are their times of flow of *equal volume* of liquids through the same capillary respectively, then

$\quad$ η_1 α $d_1$ght$_1$

$\quad$ η_2 α $d_2$ght$_2$

Hence

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

Here, usually the viscosity of given liquid is measured with respect to water whose viscosity is known very accurately at different temperatures. The SI physical unit of viscosity is the pascal-second (**Pa·s**), (i.e., kg·m−1·s−1). This means: if a fluid with a viscosity of one **Pa·s** is placed between two plates, and one plate is pushed sideways with a shear stress of one pascal, it moves a distance equal to the thickness of the layer between the plates in one second. The cgs unit for the same is the **poise** (P), (named after J. L. Marie Poiseuille). It is more commonly expressed, as **centipoise** (**cP**). [1 cP = 0.001 Pa·s]. Water at 20 °C has a viscosity of 1.0020 cP.

Procedure:

1. Note the laboratory temperature.

2. Wash the density bottle with distilled water and dry.

3. Take the weight of the empty & filled (with distilled water) specific gravity bottle (with stopper). Then, weigh the filled with specific gravity bottle unknown given liquids individually. Use the data for measuring the densities.

4. Clean and rinse the viscometer properly with distilled water. Fix the viscometer vertically on the stand and filled with specific amount (say 20ml) of mixture (every time take the same volume).

5. Time of flows was recorded for each solution (water and the given liquids).

6. Take 3 to 4 readings.

Observations:

1. Laboratory temperature=…. ^{0}C

2. Density measurement:

Weight of empty density bottle (w1) =……..g.

Weight of density bottle with water (w2) =……..g.

Weight of density bottle with castor oil (w3) =…….g.

Weight of density bottle with castor oil (w4) =…….g.

So,

Weight of water = (w2-w1) =…g.

Weight of castor oil = (w3-w1) =…g.

Weight of ground nut oil = (w4-w1) =…g

Sample	Flow times			
	t_1	t_2	t_3	Average
Water				
Castor oil				
Ground ut oil				

Calculations:

Determination of the viscosity of the liquid (η)

As discussed warlier

$$\frac{\eta_c}{\eta_w} = \frac{d_c \, t_c}{d_w \, t_w}$$

$$\eta_c = \frac{d_c \, t_c}{d_w \, t_w} \, \text{X} \, \eta_w$$

here η_c = viscosity of castor oil; η_w = viscosity of water (1.0020 cP at 20^0C)

d_c = density of castor oil; dw = density of water

t_c = flow time for castor oil; t_w = flow time for water

$$\frac{\eta_g}{\eta_w} = \frac{d_g \, t_g}{d_w \, t_w}$$

$$\eta_g = \frac{d_g \, t_g}{d_w \, t_w} \, \text{X} \, \eta_w$$

here η_c = viscosity of ground nut oil; η_w = viscosity of water

d_c = density of ground nut oil; dw = density of water

t_c = flow time for ground nut; t_w = flow time for water

Result:

The viscosity of the given castor oil with respect to water at laboratory temperature was found to be ….....…..CP.

The viscosity of the given groundnut oil with respect to water at laboratory temperature was found to be ………...CP.

EXPERIMENT-9

CORROSSION

Determination of rate of corrosion of mild steel in the absence and presence of an inhibitor

Aim: To determination of rate of corrosion of mild steel in acidic medium in the absence and presence of an inhibitor and calculate the efficiency of the inhibitor

Apparatus: 250 mL beaker, glass hooks, mild steel specimen

Chemicals: 10% HCl solution, inhibitor stock solution

Principle: Corrosion is the process of gradual decay of metal/alloys by chemical or electrochemical reaction with the environment. Corrosion of mild steel in an aqueous acidic environment is example of electrochemical corrosion and proceeds through evolution of hydrogen gas.

At anode: Fe Fe+2 + 2e

At cathode: H + + 2e- H2

In the presence of an inhibitor molecules get chemisorbed on the surface of the mild steel and form a protective film which is quite stable in the acidic environment. This protective film acts as a barrier between the metal and environment. Hence further corrosion is prevented.

Procedure :

Prepare the acidic environment in the absence and presence of an inhibitor, as shown in table1.

Table 1:

Acidic environment	10% HCl solution (Ml)	Inhibitor solution(mL)	Total volume (mL)
Absence an inhibitor	100	0	100
Presence of an inhibitor	90	0	100

Prepare the surfaces of 4 mild steel samples as to obtain smooth and bright surfaces use Emery papers. By this process you are removing the rust already formed on the surface. Clean the metal surfaces by immersing in acetone. By this you can remove any oily or greasy material on the surface. Dry in the water bath for 5 minutes, cool and weigh the samples by means of an electronic balance and record the weights.

Immerse samples 1 and 2 in the acidic environment in the absence of inhibitor and samples 3 and 4 in the acidic environment in the presence of inhibitor by means of glass hooks for 30 minutes. After which take out samples and wash in ruining water to remove the loosely held rust or other 23 corrosion products. Rinse the plates with acetone, dry in oven for 5 minutes, cool and record their weights.

Observation table:

S.NO	Weight immersion (mg)	Weight after 30 min immersion (mg)	Weight loss (mg)	Surface area of the sample dm2	Corrosion rate (mdd)
1					
2					
3					
4					

Calculation:

Corrosion rate for each sample:

Weight of the metal sample before immersion = W1 mg

Weight of the metal sample after 30 min immersion = W2 mg

Weight loss= W1-W2 mg= X mg

Surface area of the sample = Y dm2

Immersion period = Z days

Corrosion rate = X/YZ mdd (mg/dm2 /day)

Corrosion inhibitor efficiency of the inhibitor:

The average corrosion rates in the absence of inhibitor = A

The average corrosion rates in the presence of inhibitor = B

Corrosion inhibitor efficiency of the inhibitor = (A-B) X 100/A

Result: Corrosion rate of mild steel sample in the acidic medium in the absence of an inhibitor =

Corrosion inhibitor efficiency of the inhibitor (%) = ---------

ADDITIONAL EXPERIMENTS

EXPERIMENT-1

Estimation of Acetic Acid by Conductometry.

**Aim :** To determine the strength of the weak acid by titration with strong base Conductometrically.

Outcome : The student shall be able to analyze the variation of conductance values of given weak acid with addition of strong base using conductivity meter.

Chemicals Required: Sodium hydroxide, acetic acid

Apparatus: Conductivity Bridge, Conductivity cell, Burette, Beakers, Standard flask, pipette, Burette Stand.

Principle:

Before titration low initial conductance is observed due to low H^+ obtained during dissociation of weak CH_3COOH. During titration we can observe slight decrease of conductance due to consumption of H^+. During progress of titration we can observe slight increase in conductancedue to the presence of $CH3COO^-$ & Na^+ and nearly constant H^+ due to the buffer action of the produced CH_3COONa and the remaining CH_3COOH. After end point excess NaOH will lead to increase in conductance due to increasing of Na^+ and OH^-.

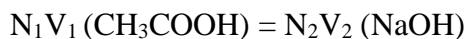$$CH_3COOH + Na^+ + OH^- \rightarrow CH_3COO^- + Na^+ + H_2O$$
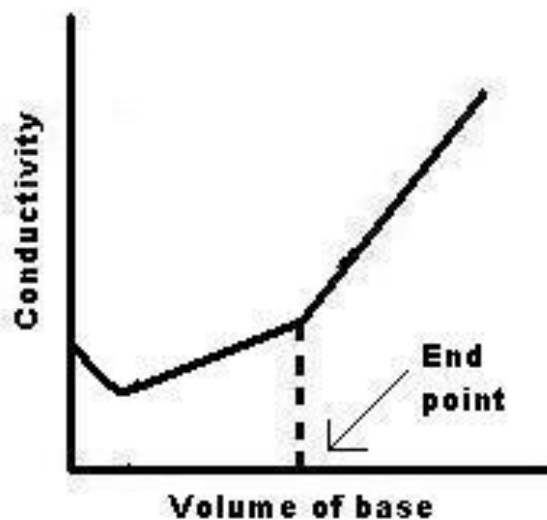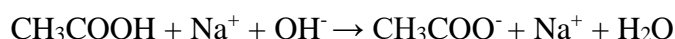
Procedure:

Prepare a standard solution of 1M NaOH. Similarly, prepare 0.1M acetic acid. 200 ml of acetic acid is taken in a 250 ml beaker. Further, the conductivity cell is washed with distilled water and rinsed with acid soln. The cell is kept in acid containing beaker and it is connected to the bridge. The conductivity of the solution is measured by adjusting the reading. NaOH solution is taken into burette and add 1 ml of solution to acid, stirred well and conductance is measured. Each time 1 ml of base is added to acid stirred well and the conductance is measured. For every instance equal numbers of values are taken on either side of the point of maximum. Repeat the procedure of addition of 1 ml NaOH and noting the conductivity of the resulting solution. Take 20-25 readings

CALCULATIONS:

Volume of unknown acid (V_1) =200 ml.

S. No	Volume of NaOH	Observed conductance
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		

FORMULA:

$$N_1V_1 \ (CH_3COOH) = N_2V_2 \ (NaOH)$$

$$N_1 = \frac{1 \ X \ V_2}{200}$$

Strength of solution = Eq. Weight X Normality

$$= 36.5 \ X \ N_1$$

$$= \text{---------------} \ g/lit$$

RESULT:

The normality of weak acid (acetic acid) determined by titrating against a strong base (NaOH) =_____N

Precautions:

1. The conductivity cell should be handled very carefully as it is very delicate.

2. Stirring should be done after each addition of the titrant.

Advantages or applications of conductance titration:

1. This method can be used to very dilute solutions.

2. Gives very accurate end points with an error of $\pm$ 0.5

3. These titrations are very useful in case of coloured solutions which cannot be titrated by ordinary volumetric method because colour change of indicator is not clear.

4. Useful of titrating weak acids against weak bases, which otherwise do not give sharp end points.

5. No keen observation is necessary near the end point since it is detected graphically.

EXPERIMENT-2

Estimation of HCl by Potentiometry

Aim: To determine the equivalence point between strong acid and strong base and to determine the normality of HCl by titrating with NaOH using potentiometer.
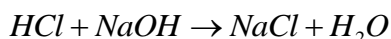
Outcome : The student shall be able to analyze the variation of EMF values of given acid with addition of strong base using potentiometer.

Chemicals required: 0.1N HCl, 1N NaOH, distilled water.

Apparatus: Potentiometer, standard cell, saturated calomel electrode, platinum electrode, beaker, burette, stirrer etc.

Principle

When a solution of strong acid (HCl) is titrated with the solution of a strong base (NaOH), the change in pH will be reflected in the change in EMF. When a small amount of alkali is added to the acid, a little change in the EMF is produced in the beginning. This change in electrode potential depends on the fraction of hydrogen ions removed. As an equivalence point is reached, the fraction of the hydrogen ions removed by constant volume of standard alkali increases rapidly, thereby causing a rapid change in the EMF. Thus if the EMF of the cell is plotted against the volume of the standard alkali added, a curve is obtained. As the change in EMF is much more rapid near the equivalent point, the exact equivalent point is obtained by differential method where, a graph of $\Delta E/\Delta V$ Vs. volume of alkali added, gives the maximum of the curve which corresponds to equivalence point of the titration.

$$HCl + NaOH \rightarrow NaCl + H_2O$$

The cell can be represented as

$$H_2(Pt)/acid\ solution//KCl(aq)/calomel\ electrode$$

Procedure

Estimation of HCl:

10 mL of 0.1N HCl is pipetted out into 100 mL beaker and saturated it with Quinhydrone and the indicator electrode (platinum electrode) is dipped. The indicator electrode and saturated calomel electrode (reference electrode) are connected to the potentiometer. The two half cells are connected by means of a salt bridge. The potentiometer is standardized and used for measuring the emf directly. 1N NaOH is taken in the burette and is added to the HCl solution.

The emf of the acid, taken in the beaker, is measured initially. Rough titration is first carried out by adding 1 mL of NaOH and the emf for each addition is measured. The emf decreases gradually and then

shows a sudden decrease in the emf. Enough readings are taken after the sudden decrease in emf. From these rough titrations, the range of end point is determined.

After finding the end point range, fair titrations are carried out by repeatedly adding 1 mL is added and emf is measured for each addition and the readings are tabulated.

Graph is plotted with volume of alkali (NaOH) along X-axis and measured emf along Y-axis. A sigmoid curve is obtained and the equivalence point is noted at the end point of intersection. To obtain a sharp end point, another graph of volume of alkali (NaOH) along X-axis and $\Delta E / \Delta V$ along Y-axis is plotted. The maxima obtained in the curve gives the accurate equivalence point.

Observations:
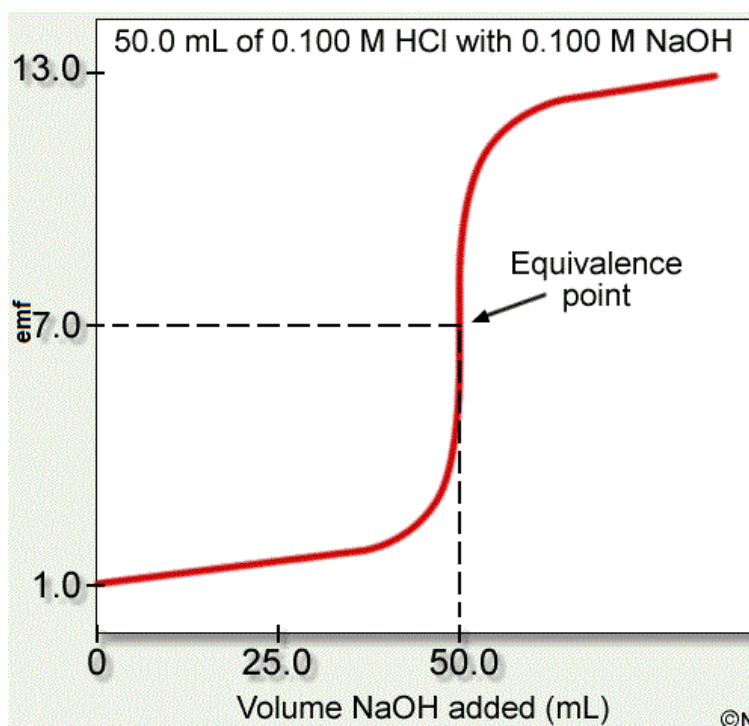
Pilot Titration:

Volume of alkali (NaOH) (mL)	EMF (mV)
0.0	
1.0	
2.0	
3.0	
4.0	
5.0	
6.0	
7.0	
8.0	
9.0	

Final Titration

Volume of alkali (mL)	EMF (mV)	$\Delta E/\Delta V$
0		
1.0		
2.0		
3.0		
4.0		
5.0		
6.0		
7.0		
8.0		
9.0		
10.0		
11.0		
12.0		
13.0		
14.0		
15.0		
16.0		

Calculations:

Strength of HCl

$Volume\ of\ acid\ taken\ in\ bea\ker, V_1 = 20\,mL$

$Strength\ of\ acid, N_1 = ?$

$Volume\ of\ NaOH\ (from\ the\ graph), V_2 = ...mL$

$Strength\ of\ NaOH, N_2 = ...$

$According\ to\ Volumetric\ Law$

$V_1 N_1 = V_2 N_2$

$N_2 = \dfrac{V_1 N_1}{V_2} N$

Amount of HCl

Amount of HCl present in the given solution=……………$(N_2)\times36.45g$

RESULT:

1. The equivalence point of potentiometric titration between strong acid Vs strong base is ... ml

2. The Normality of HCl by titrating with NaOH using potentiometer is……..N.

3. The amount of HCl present in the given solution =……….g.