ILAHIA

COLLEGE OF ENGINEERING AND TECHNOLOGY

(Affiliated to A.P.J. Abdul Kalam Kerala Technology University & Approved by AICTE)

ENGINEERING CHEMISTRY

LAB MANUAL

B.TECH. S1/S2

KERALA TECHNOLOGICAL UNIVERSITY

List of Experiments

1. Estimation of total hardness-EDTA method
2. Estimation of dissolved oxygen by Winkler's method
3. Estimation of chloride in water
4. Preparation of Urea- Formaldehyde and Phenol- Formaldehyde resin
5. Identification of compounds in the given mixture using thin layer chromatography
6. Determination of wavelength of absorption maximum and colorimetric estimation of Fe^{3+} in solution
7. Calibration of p^H meter and determination of p^h of a solution
8. Conductivity measurements of salt solutions

EXPERIMENT NUMBER 1

ESTIMATION OF HARDNESS OF WATER BY EDTA METHOD

Aim

Estimation of total hardness present in the given sample of water by EDTA method.

Principle

Hardness of water is the property by which water does not give ready and quick lather with soap. Hardness is mainly due to the presence of bicarbonates, chlorids and sulphates of calcium and magnesium ($Ca(HCO_3)_2$, $Mg(HCO_3)_2$, $CaCl_2$, $MgCl_2$, $CaSO_4$, $MgSO_4$).

EDTA (Ethylene diamine tetra acetic acid) forms colourless stable complexes with Ca^{2+} and Mg^{2+} ions in hard water. Eriochrome Black-T(EBT) a blue dye is also form complexes with Ca and Mg ions in hard water at p^H 10. These complexes are wine red in colour and less stable than EDTA complexes. When these two observations are combined we get a titration method of estimating hardness. Some hard water is taken; buffer is added to make p^H 10. Now add EDTA solution from a burette. First EDTA form complexes with the free Ca and Mg ions. Now EDTA will take away the Ca and Mg ions from the EBT complexes. When all the Ca and Mg ions are taken from EBT complex the fee blue dye is generated which is blue in colour. That is the end point. The colour of the solution changes from wine red to blue. So EBT can be used as the indicator for the titrations.

Apparatus required: Burette, Pipette, Conical flask, Beakers, Funnel

Reagents: EDTA solution, Standard hard water, given water sample, Eriochrome Black- T indicator, Buffer solution (NH_4Cl-NH_4OH).

1. Standard Hard Water: Dissolve exactly 1gm of pure anhydrous $CaCO_3$ in distilled water using dil. HCl. Make the volume to 1 litre.
   1 ml of this standard hard water = 1mg of $CaCO_3$
2. EBT indicator: Dissolve the dye in an organic solvent.
3. Buffer of p^H: Dissolving Ammonium chloride in Ammonium Hydroxide.
4. EDTA solution: Dissolve some sodium salt of EDTA in distilled water.

Procedure

1. Standardization of EDTA with standard hard water.

Pipette out 20 ml of standard hard water into a clean conical flask. Add 5 ml of buffer solution and two drops of Eriochrome Black-T (EBT), the colour of the solution turns wine red. Titrate this solution with EDTA solution taken in the burette, until the colour of the solution changes from wine red to clear blue at the end point. The final reading of the burette is noted and the titration is repeated to get concordant value. Volume of EDTA used is taken as V1 ml.

2. Estimation of total hardness of the given water sample

Pipette out 20 ml of given hard water sample into a clean conical flask. Add 5 ml of buffer solution and two drops of Eriochrome Black-T (EBT). Repeat the titrations to get concordant value. Volume of EDTA used is taken as V_2 ml.

Observations and Calculations

1. Standardization of EDTA solution

Standard hard water vs. EDTA **Indicator : EBT**

Sl.No.	Volume of standard hard water in ml	Burette reading in ml		Volume of EDTA solution in ml
		Initial	Final	
1	20			
2	20			

Concordant value of EDTA solution, V_1 = ...ml

1ml of standard hard water = 1mg of $CaCO_3$

V_1 ml of EDTA solution =20 ml of standard hard water

=20 mg of $CaCO_3$

1 ml of EDTA solution = $\frac{20}{V1}$ mg $CaCO_3$

2. Estimation of total hardness of the given water sample

Given hard water vs. EDTA **Indicator: EBT**

Sl.No.	Volume of standard hard water in ml	Burette reading in ml		Volume of EDTA solution in ml
		Initial	Final	

Concordant value of EDTA solution, V_2 = ...ml

20 ml of the given sample = V_2 ml of EDTA

= $V_2 \times \frac{20}{V1}$ mg $CaCO_3$

1 ml of given water sample = $V_2 \times \frac{20}{V1}$ mg $CaCO_3 \times \frac{1}{20}$

1000 ml or 1 L of the water sample = $\frac{V2}{V1}$ x 1000 mg $CaCO_3$

RESULT

Total hardness of the given water sample = ...ppm

EXPERIMENT NUMBER 2

ESTIMATION OF DISSOLVED OXYGEN (DO) IN WATER SAMPLE (WINKLER'S METHOD)

Aim

To determine the amount of dissolved oxygen (DO) in given water sample by Winkler's method, provided standard solution of potassium dichromate ($K_2Cr_2O_7$, 0.01N) and sodium thiosulphate ($Na_2S_2O_3.5H_2O$, N/100 or 0.01N).

Principle

Determination of dissolved oxygen is important for industrial application and aquatic life. Dissolved oxygen is also an important factor for corrosion. Dissolved oxygen is used as an indicator of the health of a water body. Any Reduction in the amount of oxygen in a body of water, caused by arises in temperature or by pollution, is harmful. The concentration of dissolved oxygen can there for be taken as a measure of the ability of water to support living things.

Solubility of oxygen in fresh water varies from 7.5 – 14.5 mg/L. The oxygen content may decrease because of the presence of organic impurities (Because of aerobic oxidation).

The Principles involved in the determination of dissolved oxygen is to bring about the oxidation of potassium iodide (KI) Iodine (I_2) with the dissolved oxygen after adding $MnSO_4$, KOH, and KI. The basic manganese oxide formed act as an oxygen carrier to enable the dissolved oxygen in the molecular form to take part in the reaction the liberated iodine is titrated against $Na_2S_2O_3.5H_2O$ (N/100), using starch as an indicator.

$$MnSO_4 + 2KOH \longrightarrow Mn(OH)_2 + K_2SO_4$$

white ppt

$$2Mn(OH)_2 + O_2 \longrightarrow 2MnO(OH)_2 \downarrow$$

Basic manganic oxide

Basic manganic oxide on acidification with conc.H_2SO_4 gives

$$MnO(OH)_2 + H_2SO_4 \longrightarrow MnSO_4 + 2H_2O + [O]$$

$$2KI + H_2SO_4 + [O] \longrightarrow K_2SO_4 + H_2O + I_2$$

The liberated iodine (I_2) is titrated against standard sodium thiosulphate ($Na_2S_2O_3.5H_2O$) solution using starch as the indicator (Starch + I_2 $\longrightarrow$ blue coloured complex)

$$I_2 + 2Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaI$$

Freshly prepared starch solution is added to the conical flask when it is nearing to the end point. The end point is the disappearance of the blue colour. Iodine release is difficult hence starch should be added only near the end point. Sodium thiosulphate can be standardized using potassium dichromate ($K_2Cr_2O_7$, 0.01N), which liberate I_2 from KI.

Apparatus required: Conical flask, Burette, Measuring jar, Beakers, Pipette, Glass rod, Iodine flask, Test tube, Dropper

Reagents: Sodium thiosulphate ($Na_2S_2O_3.5H_2O$, 0.01N), Potassium Iodide 10% (KI), Potassium dichromate ($K_2Cr_2O_7$, 0.01N), Starch, Dilute (2 N) and Conc.H_2SO_4, Manganous Sulphate ($MnSO_4$).

Procedure

1. Standardisation of Sodium thiosulphate

20 ml (V1 ml) of the standard potassium dichromate Solution ($K_2Cr_2O_7$, 0.01 N) is pipetted out into a conical flask. 20 ml of dilute sulphuric acid and 5-10ml of 10% KI are added. The liberated iodine is titrated against sodium thiosulphate ($Na_2S_2O_3.5H_2O$) taken in the burette till the colour changes to yellowish green (straw yellow). At this stage 1ml of freshly prepared starch is added as indicator to the conical flask when it is nearing the end point. The titration is the continued until the colour change from blue to light green which is the end point. The titration is repeated to get concordant value. Concordant volume of $Na_2S_2O_3.5H_2O$ is noted as V_2 ml.

2. Estimation of Dissolved Oxygen

250 ml iodine flask is filled with the given water sample up to the neck. Add 2 ml of $MnSO_4$ solution to the sample using a pipette well below the surface of the solution (some over flow will occur). Similarly introduce 2ml of alkaline potassium iodide solution (KOH & KI) and then stopper the iodine flask. Iodine flask is shaken vigorously to help dissolved

oxygen to react and kept for about 20 minutes for the brown precipitate to get settle down. When the precipitate has settled at least 3 cm below the stopper, introduce few ml of conc. H_2SO_4 by carefully running the acid down the side of the flask. Replace the stopper and carefully mix until the precipitate disappears and a clear yellow solution is obtained due to liberation of I_2. If the precipitate is not dissolved, add more acid. A magnetic stirrer is helpful here. Allow the mixture to stand for 5 minutes. 100 ml (V_3 ml) of this solution is measured in a measuring jar and transferred to a conical flask. This is titrated against standard sodium thiosulphate ($Na_2S_2O_3.5H_2O$) in the burette until the colour becomes pale yellow. 1 ml starch is added as indicator near the end point so that the solution turns blue. Continue titration till blue colour disappears. The titration is repeated to get concordant value.

Observation and Calculations

1. Standardisation of $Na_2S_2O_3.5H_2O$ with $K_2Cr_2O_7$ solution

Std.$K_2Cr_2O_7$ vs. $Na_2S_2O_3.5H_2O$ **Indicator: Starch**

Sl.No.	Volume of $K_2Cr_2O_7$ in ml (V_1)	Burette Reading		Volume of $Na_2S_2O_3.5H_2O$ in ml (V_2)
		Initial	Final	
1	20			
2	20			
3	20			
4	20			

Concordant volume of $Na_2S_2O_3.5H_2O$ solution, V_2 =.....ml

Volume of $K_2Cr_2O_7$ in ml V_1 = 20ml

Normality of $K_2Cr_2O_7$ N_1 = 0.01 N

Volume of $Na_2S_2O_3$ V_2 = ...ml

Normality of $Na_2S_2O_3$ N_2 ,

$$N_2 = \frac{N1V1}{V2} = \frac{0.01X20}{V2}$$

Normality of $Na_2S_2O_3$ N_2 = ...N

Estimation of Dissolved Oxygen (DO)

Given water sample Vs. $Na_2S_2O_3.5H_2O$ **Indicator: Starch**

Sl.No.	Volume of Water sample in ml (V_3)	Burette Reading		Volume of $Na_2S_2O_3.5H_2O$ in ml (V_{2a})
		Initial	Final	
1	100			
2	100			
3	100			
4	100			

Concordant volume of $Na_2S_2O_3.5H_2O$ solution, V_{2a} =.....ml

Volume of $Na_2S_2O_3$ V_{2a} = ...ml

Normality of $Na_2S_2O_3$ N_2 = ...N

Volume of the given water sample V_3 = 100 ml

Normality of the given water sample N_3,

$$N_3 = \frac{N2V2}{V3} = \frac{N2\ XV2a}{100}$$

Normality of the given water sample N_3 = ... N

Amount of dissolved oxygen in 1lit of the given water sample in mg/L or ppm

= N_3x Eq.Wt.of oxygen x1000

(Eq.Wt. of oxygen =8)

=.....mg/L or ..... ppm

RESULT

Amount of DO present in the given water sample =.....mg/L or ..... ppm

EXPERIMENT NUMBER 3

ESTIMATION OF CHLORIDE IN A GIVEN WATER SAMPLE BY ARGENTOMETRIC METHOD (MOHR'S METHOD)

Aim

To determine the chloride content in the given water sample by Mohr's Method

Principle

Chloride ions in a water sample (neutral or slightly alkaline) can be determined by titrating it against standard silver nitrate ($AgNO_3$) solution using potassium chromate (K_2CrO_4) as an indicator. The PH should be in between 7-8.

This method is based on the precipitation titration in which silver nitrate solution is released from the burette to the water sample which contains chloride icon and indicator. The silver ions (from silver nitrate solution) react with chloride icons (from water sample) and to form white precipitate of silver chloride. At the end point the colour changes from bright yellow to faint reddish brown colour due to the formation of silver chromate.

$$Ag^+ + Cl^- \rightleftharpoons AgCl$$

White precipitate

$$2Ag^+ + CrO_4^{2-} \rightleftharpoons Ag_2CrO_4$$

Red precipitate

Apparatus required : Burette, Pipette, Conical flask, Measuring jar, Beakers, Dropper

Reagents : Standard silver nitrate solution ($AgNO_3$, N/100), 5% potassium chromate indicator solution (K_2CrO_4)

Procedure

1. Titration with the Blank solution

Take 50 ml of distilled water in a conical flask. Measure p^H and add 3-4 drops of potassium chromate indicator solution (K_2CrO_3). Slowly add standard silver nitrate ($AgNO_3$, N/100) solution from the burette and shake the solution well. The titration is then continued until the colour change from light yellow to red which is the end point. The titration is repeated to get concordant value. Concordant volume of $AgNO_3$ is noted as V_1 ml. The blank correction for the indication should be subtracted from the volume of the titrant obtained after titrating the sample solution given in step 2.

2. Estimation of chloride ion in the given water sample.

Transfer 50 ml of the given water sample in a conical flask. Measure the p^H and add 3-4 drop of indicator potassium chromate solution (K_2CrO_4). Slowly add standard silver nitrate ($AgNO_3$, N/100) solution from burette and shake the solution well. The titration is then continued until the end point is obtained. The end point is the colour change from light yellow to red and the red colour persists. The titration is repeated to get concordant value. Concordant volume of $AgNO_3$ is noted as V_2 ml.

Observation and Calculations

1. Titration with the blank solution

Distilled water vs. $AgNO_3$ **Indicator : K_2CrO_4**

Sl.No.	Volume of distilled water in ml	Burette reading in ml		Volume of $AgNO_3$ solution in ml (V_1)
		Initial	Final	
1	50			
2	50			
3	50			
4	50			

Concordant volume of $AgNO_3$ solution,V_1 =.....ml

2. Estimation of chloride ion in the given water sample

Given water sample vs. $AgNO_3$ **Indicator : K_2CrO_4**

Sl.No.	Volume of given water sample in ml	Burette reading in ml		Volume of $AgNO_3$ solution in ml (V_2)
		Initial	Final	
1	50			
2	50			
3	50			
4	50			

Concordant volume of $AgNO_3$ solution,V_2 =.....ml

Normality of standard $AgNO_3$ solution, N_A = N/100 = 0.01N

Volume of standard $AgNO_3$ solution, V_A = $[V_2-V_1]$

= .....ml

Volume of given water sample, V_W =50 ml

Normality of given water sample, N_W can be calculated from the normality formula,

$$N_A \times V_A = N_W \times V_W$$

$$N_A \times V_A = N_W \times 50$$

$$N_W = N_A \times V_A/50 = ...N$$

Amount of chloride ions = N_W x eq. Wt. of Chlorine = N_W x 35.45g/L

=.....g/L

Amount of chloride ions in ppm = ...x 1000 mg/L or ppm = ...mg/L or ppm

RESULT

The amount of chloride ion in the given water sample is ..... mg/L or ppm

EXPERIMENT NUMBER 4A

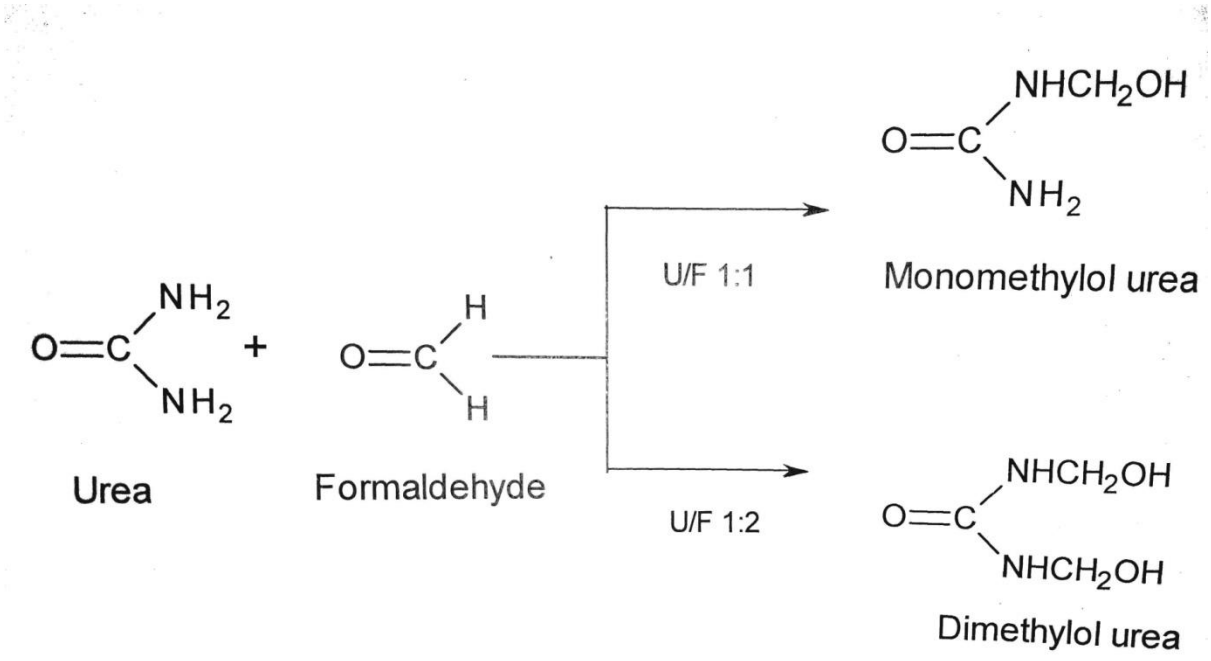
PREPARATION OF UREA-FORMALDEHYDE RESIN

Aim

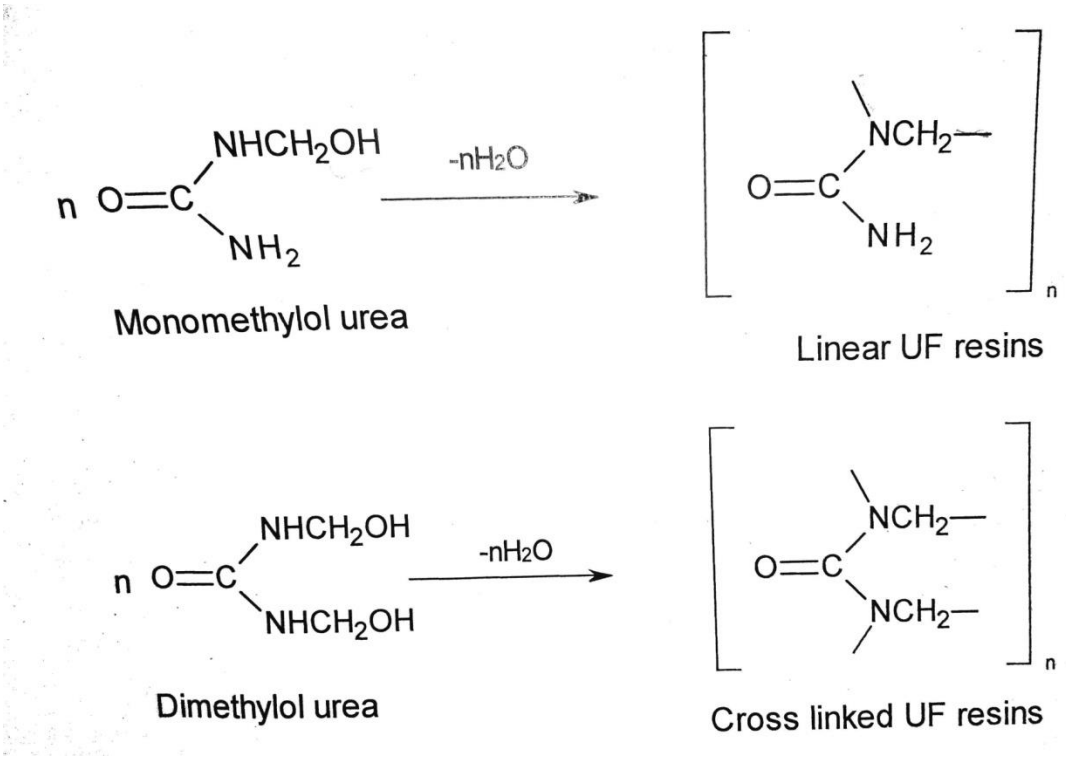
To prepare Urea-Formaldehyde resin (UF Resin)

Principle

Urea-Formaldehyde resin is prepared by condensation reaction between urea and formaldehyde in neutral or acidic medium. It is a thermosetting resin. The first product formed during the formation of resin is monomethylol and dimethylol ureas, which undergo further condensation reaction to give linear, partially cross-linked or fully cross-linked polymer.



Several molecules of methylol urea derivatives condense with loss of water molecules to form a linear or highly cross linked urea formaldehyde resin.



Apparatus required: Glass rod, Beakers, Funnel, Filter paper

Reagents: Urea (2g), 40% formaldehyde solution, conc.H_2SO_4

Procedure

Place 5 ml of 40% formaldehyde solution in a 250 ml beaker. Add about 2 grams of urea while stirring until a structured solution is obtained. Add a few drops (2-3 drops) of conc. H_2SO_4 with stirring. Stir the solution cautiously during the addition. All of a sudden a voluminous white solid mass appears in the beaker. When the reaction is complete, wash the residue with distilled water to remove the acid. Dry the product in a filter paper. The solid resin formed is powdered and appearance and yield are noted.

RESULT

Appearance: White flexible solid

Weight of Urea-Formaldehyde resin formed =……g

EXPERIMENT NUMBER 4B

PREPARATION OF PHENOL-FORMALDEHYDE RESIN

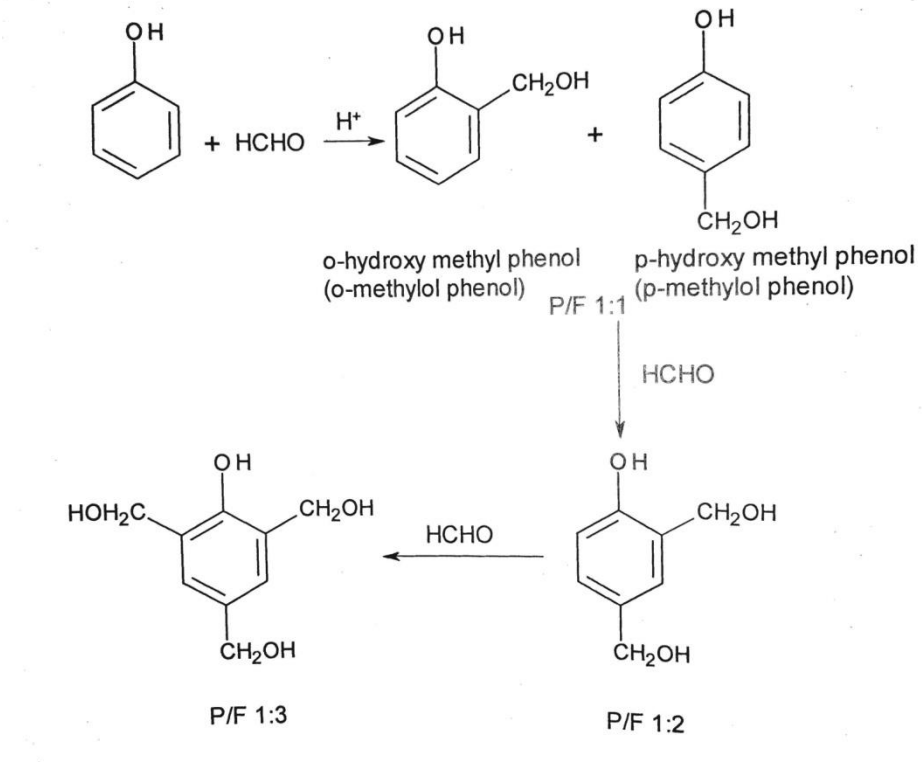
Aim

To Prepare Phenol-Formaldehyde resin (Bakelite or PF Resin)

Principle

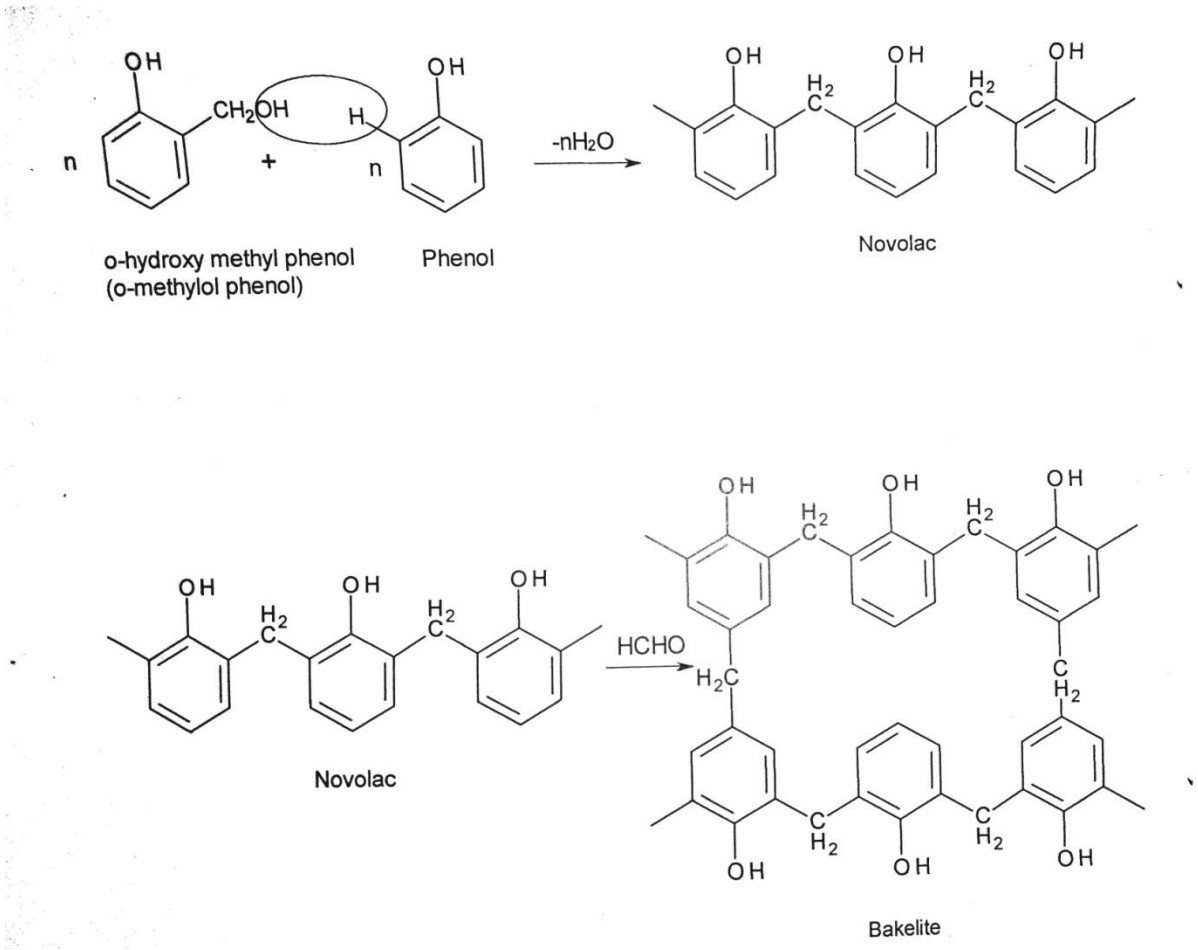
Phenol-Formaldehyde (also called Bakelite) is the oldest synthetic polymer. These are thermosetting polymers, which on heating change irreversibly into hard, rigged and infusible materials.

It is prepared by the condensation reaction of phenol and formaldehyde in presence of acid or base catalyst. In presence of acid analyst, the reaction starts with initial formation of *o*- and or *p*- hydroxymethyl phenol (methylol) derivatives, which further react with phenol to form compound having rings joined to each other through $-CH_2$ groups. The initial product is linear polymer called Novolac, which is used in paints. Novolac on heating with formaldehyde undergoes cross linking to form infusible solid mass called Bakelite.



The phenol formaldehyde derivatives react among themselves or with phenol to give a linear polymer or a higher cross linker polymer.

Linear Polymer –Novolac



Apparatus required: Glass rod, Beakers, Funnel, Filter paper, Dropper, Measuring cylinder

Reagents: Glacial acetic acid (17.4N, 99.5%), formaldehyde solution, phenol, Conc.HCI

Procedure

Place 5 ml of glacial acetic acid and 2-2.5 ml of 40% formaldehyde solution in 500 ml beaker and add 2 g of phenol. Wrap a cloth or towel loosely round the beaker. Add a few ml of Conc. HCI into the mixture carefully. Warm the beaker slightly to melt the phenol then shake it to obtain a homogeneous solution. Heat the solution slowly with constant stirring for

5 minutes. Within 5 minutes, a large mass of pink plastic is formed. The residue obtained is washed several times with distilled water. The product is then filtered and dried. Calculate the yield of the product. Heat the mixture carefully with a low flame as this is an exothermic reaction and too much heat will cause spattering and, potentially, could cause your breaker to break.

Note: The reaction is sometimes vigorous and it is better to stand a few feet away from the beaker while adding the HCI and until the reaction is complete. The experiment should be preferably carried out in a fume cupboard. Most phenols are harmful if inhaled, ingested or absorbed through skin. They cause severe irritation or damage to skin and eyes. Phenols are carcinogenic, should not inhale its vapour, wear gloves and avoid contact.

RESULT

Appearance: Pink solid

Weight of phenol-formaldehyde resin formed =…….g

EXPERIMENT NUMBER 5

IDENTIFICATION OF COMPOUNDS IN THE GIVEN MIXTURE USING THIN LAYER CHROMATOGRAPHY

Aim

Separation of components from the given mixture and determination of R_f value by using thin layer chromatography.

Principle

Thin-layer chromatography is a chromatographic technique in which compounds are separated on a thin layer of adsorbent material. It is also used to check the purity of a mixture and to monitor the progress of a reaction. Adsorption is the principle of thin layer chromatography. So the stationery phase is solid and mobile phase is liquid or gas. Separation is based on the adsorbing capacity of components.

TLC is performed on a sheet of glass, plastic, or aluminium foil, which is coated with a thin layer of adsorbent material, usually silica gel, aluminium oxide (alumina), or cellulose. This layer of adsorbent is known as the stationary phase.

After the sample has been applied on the plate, a solvent or solvent mixture (known as the mobile phase) is drawn up the plate via capillary action. Because different analytes ascend the TLC plate at different rates, separation is achieved. The mobile phase has different properties from the stationary phase.

The rate at which a compound moves in respect to the solvent front, R_f, is characteristic of that compound under standard condition. The R_f value can be calculated by using the following equation:

$$R_f = \frac{\text{Distance moved by the component}}{\text{Distance moved by the solvent front}}$$

Where, R_f is called retention factor.

Procedure

Given a mixture of two components. Mark lines on the TLC plate 1 cm from both sides. A small drop of the given mixture is spotted on the centre of the line on one end using a capillary tube. Take a mixture of hexane and ethyl acetate in the ratio 1:1 in a TLC chamber to a depth of less than 1 cm. Place the plate in the chamber such a way that the spot of the sample do not touch the surface of the eluent in the chamber and the lid is closed. The solvent moves up the plate by capillary action, meets the sample mixture and carries it up the plate (elutes the sample). The plate should be removed from the chamber before the solvent front reaches the top line on the stationary phase (continuation of the elution will give a misleading result) and dried.TLC plate is then placed under UV light. Components are separated and mark the spots and solvent front. Measure the distance that the components and solvent front has travelled and find the R_f value.

Calculation

Distance moved by component 1 from the spotted line = cm

Distance moved by component 2 from the spotted line = cm

Distance moved by the solvent front = cm

R_f value of component 1 = $\frac{\text{Distance moved from the spotted line by component 1}}{\text{Distance moved by the solvent front}}$ =

R_f value of component 2 = $\frac{\text{Distance moved from the spotted line by the component 2}}{\text{Distance moved by the solvent front}}$ =

RESULT

Components are separated and got two spots.

R_f value of component 1 =

R_f value of component 2 =

EXPERIMENT NUMBER 6

DETERMINATION OF WAVELENGTH OF ABSORPTION MAXIMUM AND COLORIMETRIC ESTIMATION OF Fe^{3+} IN SOLUTION

Aim

To determine the wavelength of absorption maximum and estimate the concentration of Fe^{3+} ion in the given solution colorimetrically.

Principle

Colorimetry is concerned with the visible region of the spectrum and it is the science of quantitative estimation of colour and is frequently used in biochemical investigations. The quantity of light that is absorbed by a solution depends on the concentration of the dissolved solute that is absorbing the light. By measuring the amount of light absorbed, we can find the concentration of the solutions. The quantity of a substance in a mixture can be determined colorimetrically by allowing the substance to bind with color forming chromogens. The difference in color is directly related to the difference in the absorption of light. To make the presence of iron visible in solution, thiocyanate(SCN^-) ions are added. These react with Fe^{3+} ions to form blood red coloured complex.

$$Fe^{3+}(aq)+6SCN^-(aq). \longrightarrow [Fe(SCN)_6]^{3-}(aq)$$

By comparing the intensity of the color of this solution with the color of a series of standard solutions, with known Fe^{3+} concentrations, the concentration of Fe^{3+} in the unknown solution may be determined.

The colorimetric estimation is based on the Beer-Lambert's Law. It states that when a monochromatic radiation is passed through a solution, the decrease in intensity with thickness of the solution is directly proportional to the intensity of the incident radiation as well as the concentration of the solution. According to Beer-Lambert's law

$$\mathbf{\log I_0/I = A = \varepsilon cl}$$

Where,

I = Intensity of transmitted light. I_0= Intensity of incident light

ε = molar absorptivity coefficient c = concentration

l = thickness of the solution A = Absorbance

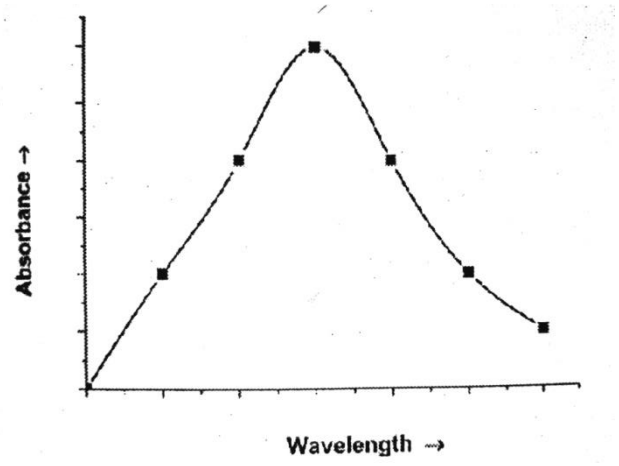
The amount of light that passes through a solution is known as transmittance. Transmittance can be expressed as the ratio of the intensity of transmittance light IT and the intensity of the incident light I_0.

Transmittance $\mathbf{T = I_0/I}$

$\mathbf{\log I_0/I}$ is called the **absorbance (**also called Optical Density or percent transmittance) represented as **A** and is measured from the colorimeter. The relative amount of light of a given wavelength that is absorbed by a solution is called (A).,where as the fraction of light entering the solution that passes through is called percent transmittance(%T).

1. Wavelength of Absorption Maximum Using Spectrophotometer

Wavelentgth of absorption maximum (λ_{max}) has to be determined before the estimation of iron in the given unknown solution. Using the blank as a reference and any one of the standard solution of iron prepared, measure the absorbance at different wavelengths in the range 350-650 nm usually in intervals of 20 nm. In the region of maximum absorbance the interval should be 5 nm. Plot the absorbance vs. wavelength and connect the points to form a smooth curve.

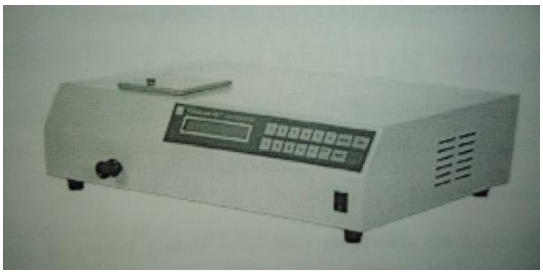


Description of the Spectrophotometer

Spectrophotometer consists of the following parts:

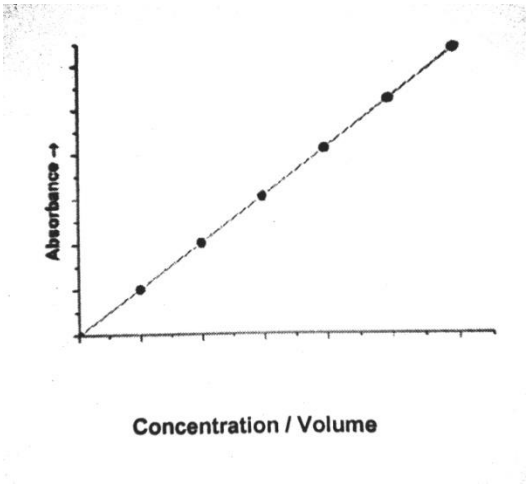
1. Source (UV and visible)
2. Wavelength selector(monochromator)
3. Sample containers
4. Detector

5. Signal processor and readout



2. Estimation of iron using colorimeter/ Spectrophotometer

Several standard solutions of known concentrations are prepared and absorbance is determined for each concentration using colorimeter. Measure the absorbance of each of the standard solutions at the selected wavelength (wavelength of absorption maximum). Absorbance is then plotted against concentration/volume of the standard solution to get the calibration curve (Beer-Lambert's plot) and it will be a straight line passing through the origin. A solution of unknown concentration is placed in the colorimeter and its absorbance is determined. The concentration/ volume of the unknown solution can be determined by interpolating its absorbance on the Beer-Lambert's plot (calibration curve). Hence the weight of iron in the given sample can be calculated.



In colorimetric estimation, it is necessary to prepare a definite volume of the **blank** solution (distilled water) to calibrate the colorimeter, test solution (sample solution with unknown solution) and a series of **standard** solutions (sample solutions with known concentration).

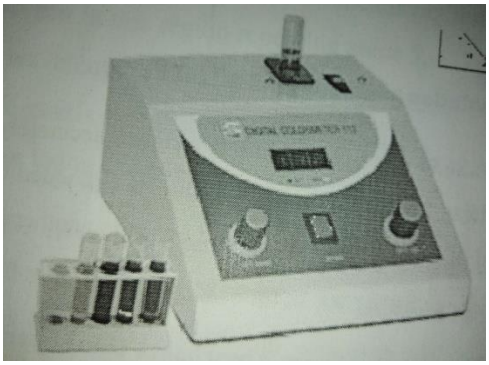
Conditions for satisfactory analysis are

1. The colour intensity should increase with concentration of the substance.
2. Colour produced should be sufficiently stable to permit an accurate measurement

3. The solution must be free from precipitate

Advantages are

1. At low concentration, colorimetric method give more accurate results than volumetric analysis.
2. Simple procedure



Description of the Colorimeter

Colorimeter consists of the following parts:

1. Light source (LED)
2. Filter (the device that selects the desired wavelength)
3. Cuvette chamber (the transmitted light passes through compartment where the coloured solutions are kept in cuvette, made of glass or disposable plastic (polystyrene))
4. Detector or photodiode (this is a photosensitive element that converts light into electrical signals)
5. Galvanometer(measures electrical signal quantitatively)

Light from the LED light source is passed through a cuvette(containing a solution of the sample). Some of the incoming light is absorbed by the solution. As a result, light of lower intensity strikes the photodiode.

Apparatus required: Spectrophotometer, Colorimeter, cuvette, measuring cylinder, standard flask, beakers, conical flask, pipette, test tubes

Reagents: Ferricalum (Ferric ammonium Sulphate ($FeNH_4(SO_4)_2.12H_2O$)), Conc. HCl (11.6N), HCl (5N), ammonium thiocyanate (20%)

Procedure:

1. **Preparation of standard solution containing 0.1g/L of Fe^{3+}**

w g (approximately **0.8634 g)** of Ferric alum is weighed accurately in to a 100 ml standard flask (0.8634g of Ferric alum contains 0.1 g of iron). 10 ml conc. HCl is added to prevent hydrolysis. It is then made up to 100 ml and 10 ml of this solution is pippetted out into another standard flask and then made to 100 ml.

2. **Measurement of Wavelength of Maximum Absorbance Using Spectrophotometer**

Blank solution is used as the reference. **6 ml** of the Ferric alum solution prepared is accurately transferred into a 100 ml standard flask. 5 ml of **5N HCl** and **20% ammonium thiocyanate** solution are added and make up the solution to 100 ml with distilled water. The absorbance of the solution is measured using the spectrophotometer. Measure the absorbance at different wavelengths in the range 350-650 nm usually in intervals of 20 nm. In the region of maximum absorbance the interval should 5 nm. Plot the absorbance vs. wavelength and connect the points to form a smooth curve.

Steps for operating spectrophotometer

1. Select a dropper bottle containing one of the available coloured solutions.
2. Hold cuvette by the rough sides. Rinse the cuvette with a small amount of the coloured solution and then fill the cuvette ¾ full.
3. Fill the second cuvette with distilled water to be used as a blank.
4. Press A/T/C Button on the Spectrophotometer. Select the absorbance.
5. Press the **nm** arrow up or down and select 350 **nm**.
6. Insert the blank into the cell holder and close the door. Position the cell so that the light passes through clear walls.
7. Press **0 ABS/100% T** to set the blank to 0 A.
8. Remove the blank and insert one of the standard solutions into the cell holder. Record the absorbance on the data sheet.
9. Reset the wavelength to 375 nm and repeat the steps 6,7,8
10. Repeat steps 5, 6, 7 and 8.Record absorbance at every 20 nm using this technique until you reach 650 nm.
11. Locate the 50 mm region in which the absorbance is highest and record the absorbance at the interval of 5nm in this region repeating steps 5, 6 ,7 and 8.
12. Determine the wavelength of maximum absorbance by plotting a graph with wavelength on the x-axis and absorbance on the y-axis.

3. Measurement of Absorbance Using Colorimeter

2,4,6,8 and **10 ml** of the Ferric alum solution (standard solutions) prepared are accurately transferred in to a five different 100 ml standard flasks l of **5N HCl** and **20% ammonium thiocyanate** solution are added and make up the solution to 100 ml with distilled water. The optical density or absorbance of the solutions are measured using the Colorimeter, by setting the wavelength at 480 nm using a blank solution. The measured values are plotted against the volumes of the standard solutions to obtain the calibration curve.

The given unknown solution is made up to 100 ml. Then **5** and **7 ml** of this solution are accurately transferred to two different 100 ml standard flasks. 5 ml of 5Nn HCl and 20 % ammonium thiocyanate solution are added and make up the solution to 100 ml with distilled water. Absorbance of the solution is measured as before using colorimeter. Using the absorbance, the volume of the standard solution corresponding to the volume of unknown solution can be determined from the calibration curve. From the volume, mass of iron in the unknown solution can be calculated.

Steps for operating the photoelectric colorimeter

1. Choose the glass filter recommended in the procedure and insert in the filter.
2. Fill the cuvettes with the blank solution to about three-fourth and place it in the cuvette slot.
3. Switch on the instrument and allow it to warm up for about 4-5 minutes.
4. Adjust to zero absorbance or optical density.
5. Rinse the cuvette and fill the test solution in another cuvette and read the absorbance or optical density.
6. Take the standard solution of varying concentration and note down the absorbance as S1, S2, S3, S4, S5, and so on.
7. A graph is plotted taking volume of the standard solution vs. the absorbance.
8. From the graph, the volume of the unknown solution can be determined.

Observations and Calculations

Colour of the solution .....

Mass of ferric alum taken = w g

Mass of Fe^{3+} in 482.25 Ferric alum = 55.85 g

.08634 g of ferric alum contains 0.1 g of iron

Mass of iron in the ferric alum taken= $\frac{0.1\ x\ w}{0.8634}$ g

w g of alum is made up to 100 ml

1 ml of this solution contains = $\frac{0.1\ x\ w}{0.8634\ x\ 100}$ g of iron

10 ml of this solution is made up to 100 ml to prepare the standard solution

1 ml of the standard solution contains = $\frac{0.1\ x\ w}{0.8634\ x\ 100\ x\ 10}$ g of iron

1. Determination of wavelength of maximum absorbance (λmax)

Wavelength (nm)	**Absorbance**	**Wavelength (nm)**	**Absorbance**
350		520	
370		540	
390		560	
420		580	
440		600	
460		620	
480		640	
500		650	

Wavelength of absorption maximum (λmax) = ... nm

2. Estimation of Fe^{3+} ion using colorimeter

Volume of Ferric alum in ml	**Absorbance**
2	
4	
6	
8	
10	
Unknown	
5	
7	

Volume of the unknown solution used = V_1 ml

Volume corresponding to standard solution from the calibration curve = V_2 ml

V_1 ml of unknown solution is equivalent to V_2 ml of standard solution

Mass of iron corresponding to V_2 ml of standard solution $= \frac{0.1\ x\ w}{0.8634}$ g = x g

Therefore mass of iron corresponding to V_1 ml of the unknown solution = x g

Mass of iron in 100 ml of the given solution $= \frac{x*100}{V1}$

Mass of iron in 1000ml of the given solution $= \frac{x*100*10}{V1}$ g

RESULT

The concentration of Fe^{3+} ion in the given solution is .....g/L

EXPERIMENT NUMBER 7

CALIBRATION OF P^H METER AND DETERMINATION OF P^H OF A SOLUTION

Aim

To calibrate the P^H meter with buffer solutions having P^H 4, and to determine the P^H of a solution

Principle

A P^H meter is an electronic device used for measuring the P^H of a solution, which is expressed as the negative logarithm of the hydrogen ion concentration $P^H = - \log (H^+)$. The P^H value indicates whether a solution is acidic or basic. If the P^H =0 it is very acidic, P^H =14 is very alkaline and P^H =7 is neutral.

The commonly used electrodes to measure P^H of a solution are Hydrogen electrode, Quinhydrone electrode and glass electrode. The P^H electrode commonly used in a P^H measurement is combined glass electrode. It consists of sensing half cell and reference half cell, together from an electrode system. The sensing half cell is a thin P^H sensitive semi permeable membrane, separating two solutions viz., the outer solution (the sample to be analysed) and the internal solution (enclosed inside the glass membrane and has a known P^H value). An electrical potential is developed inside and another electrical potential is developed outside, difference in potential is measured and is given as the P^H of the sample.

The response of a P^H electrode is defined by the Nernst equation:

$$\textit{Electrode Response} = E^0 - \frac{2.303RT}{nF} \times P^H$$

Where, E^0 = Standard electrode potential, R = gas constant, F = Faraday constant,

T = temperature in Kelvin and n = number electrons involved in the reaction.

Apparatus required: P^H meter, combined electrode, Beaker, Glass rode, Measuring jar

Reagents: Buffer solutions of P^H 4, P^H 7 and P^H 9.2, unknown solution

Procedure

1. Calibration of P^H meter

All P^H electrodes require calibration from time to time. Ensure that P^H meter is on and to obtain high precision of measurement, let the P^H meter to warm up for 30 minutes. Rinse the P^H electrode with distilled water and dry the outside of the electrode with a paper towel. Select the calibration mode on the P^H meter. Place electrode in to 20 ml of P^H 7 buffer solution making sure that the junction (located on the bottom side of the electrode) is wet (only 1 to 2 inches). Stir the Solution with a glass rod or use a magnetic stirrer. When the value (7.0) in the display has stopped changing, press the confirm key. Place 20 ml of another buffer (usually P^H 4) solution. The user can accept this value or change in to the desired second calibration point by adjusting proper key. Rinse the electrode as before in distilled water and dry. Place the electrode in the in the next buffer (usually P^H 9.2) solution selected. Wait for the meter to accept the value and press the confirm key. All P^H buffer solutions above P^H 7 are less stable and have limited life. These high P^H buffers will more readily absorb CO_2 from the atmosphere and will typically change to a lower P^H value when left open. For this reason a P^H 4 buffer solution is recommended to perform a reliable two point calibration. Also the buffers should bracket the desired P^H range.

2. Determination of P^H a solution

After calibration, take 20 ml of unknown sample in a glass Beaker to measure the P^H. Rinse the electrode and immerse it in the given solution. Wait for 1 minute to stable the reading. Note down the P^H displayed. Rinse the electrode with distilled water and move it to the storage beaker.

RESULT

P^H of the given solution =.....

EXPERIMENT NUMBER 8

CONDUCTIVITY MEASUREMENTS OF SALT SOLUTIONS

Aim

To measure the conductivity of salt solutions with a series of unknown concentration and to calculate the concentration of the give3n unknown solution by using conductivity meter.

Principle

Electrolytic conductivity of a solution is defined as the ability or power to conduct electricity. Solutions of electrolyte conduct electricity by the migration of ions under the influence of an electric field. Like a metallic conductor, they obey Ohm's law. Exceptions to this law occur only under abnormal conditions (very high voltage or high frequency current).

According to Ohm's law, the current flowing through a conductor is inversely proportional to the resistance R (Ohms) of the conductor.

$$I=V/R$$

Resistance of a sample of length l and area of cross section A is given by

$$R \alpha \ l/A \text{ or } R= \rho l/A$$

Conductance C is the reciprocal of resistance Rand its unit is mhos (reciprocal of ohms) or as Siemens.

ρ is the specific resistance or resistivity and it depends on the nature of the conductor. At a given temperature, the conductivity of an electrolyte depends on type of ionsa and on their concentration.

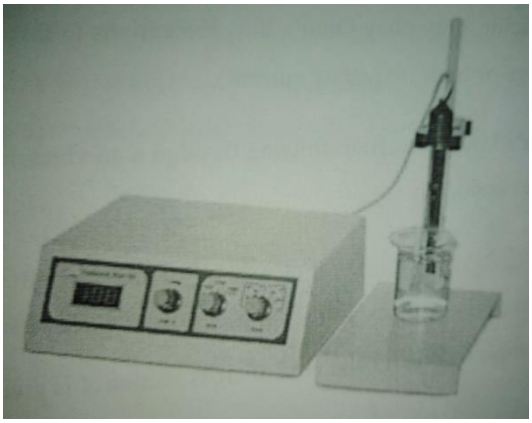
The conductivity is directly linked the concentration of ions and their mobility.The conductivity depends on the value of P^H, temperature and on the amount of dissolved CO_2. Specific conductance K is the inverse of specific resistance or resistivity (SI unit is Sm^{-1}).

$$K = Cxl /A$$

When l= 1cm and A =1cm^2, K = C, The specific conductance or conductivity of an electrolyte is defined as the conductance of a solution contained between two parallel electrodes of unit cross sectional area which are kept at unit distance apart (unit volume).

l/A is the cell constant and its unit is cm^{-1}

$$\text{Cell constant} = \frac{Specific\ conductance\ (\kappa)}{Observed\ cnductance(C)}$$



***Equivalent conductance*:-** It is defined as the conducting power of all the ions produced by dissolving one gram equivalent of an electrolyte in a solution. The equivalent conductance of an electrolyte is given by

$$\lambda eq = \frac{1000\kappa}{N} \quad \text{SI unit is } Scm^2eq^{-1}$$

Molar conductance:- It is defined as the conducting power of all the ions produced by dissolving one gram mole o an electrolyte in a solution. The molar conductance o an electrolyte is given by

$$\lambda eq = \frac{1000\kappa}{M} \quad \text{SI unit is } Scm^2mol^{-1}$$

At first conductivity meter has to be calibrated. For this purpose, a standard KCl solution whose conductivity is known is used. Conductivity can be adjusted to the known value by using the calibration knob. Then the given electrolyte is taken in the conductivity cell and its conductance is measured. A voltage is applied between the two electrodes in the probe, which is immersed in the sample solution. Equivalent conductance is calculated using the equation given above.

Apparatus required: Conductivity meter, magnetic stirrer, standard flask, measuring jar, 250 ml beaker, Funnel

Reagents: KCl solution, given electrolyte (NaCl or KCl)

Procedure

1. Calibration of conductivity meter

Switch on the instrument and wait for a half a minute. Take 50 ml o 0.1 N KCl solution in the beaker. Stir the solution. Place the electrodes in the solution. Select the calibration button and adjust the conductivity of 0.1 N KCl solution to 14.12 mmhos/cm at 30^0C. After calibration, calibration button should not be disturbed until the experiment is completed.

2. Determination of equivalent conductance of the given NaCl solution

Prepare a standard solution of NaCl by weighing out approximately 0.585 g of NaCl and dissolve it in 1000 ml distilled water (concentration is 0.1 M). Measure out accurately 3, 6, 9, 12, 15, 18 ml of this solution to different standard flasks and make up to 100 ml (the concentration ranges from .03 M to 0.18 M). Conductivity cell is filled with 50 ml of NaCl solution with different concentration, and the conductance is measured by using the conductivity meter. The experiment is repeated two times and the average value is taken. The given unknown solution is made up to100 ml with distilled water and the conductance is measured as before. A graph is plotted with concentration on the X-axis and the measured conductance on the Y-axis. From the straight line graph, the concentration of the unknown solution can be determined.

Observation and Calculation

Determination of equivalent conductance of the given solution

Sl. No.	Concentration of the electrolyte (NaCl)	Observed conductance		Observed conductance (Average)
		Trial 1	Trail 2	
1	0.03 M			
2	0.06 M			
3	0.09 M			
4	0.12 M			
5	0.15 M			
6	0.18 M			
7	unknown			

Observed conductance of the given unknown solution, C = ...S

Concentration of the given unknown solution = ...M

RESULT

Concentration of the given unknown solution =.....M