EXPERIMENT:-1

AIM: - Determination of Ca^{2+} and Mg^{2+} hardness of water sample by using EDTA solution.

CHEMICALS USED: - 0.02N $CaCO_3$ solution (standard hard water), Approx. 0.2N EDTA solution, Eriochrome Black-T indicator (EBT), NH_4OH- NH_4Cl Buffer solution (pH~ 9-10), Water sample (Unknown Hardness)

APPARATUS REQUIRED: - Conical flask, burette, pipette, measuring cylinder, funnel, filter paper, dropper, beaker (100mL)

PRINCIPLE: - Hardness is defined as the soap consuming capacity of water. Bicarbonates of Ca and Mg cause temporary hardness while chlorides, sulphates and carbonates of Ca and Mg contribute for permanent hardness. Temporary hardness can be removed by simple physical process like boiling but for removing the permanent hardness chemical treatment is required. Hard water may have a number of disadvantages thus softening of water is very essential for industrial and domestic uses.

$$\mathbf{2HCO_3^- \xrightarrow{Boiling} CO_2 + H_2O + O_2}$$

Ca^{2+} and Mg^{2+} ions form complexes easily with EDTA (Ethylene diamine tetra acetic acid), which is the basic principle of this experiment. The Ca^{+} and Mg^{+2} ions present in water are titrated with EDTA solution using Eriochrome Black-T (EBT) as indicator. Since the action of the indicator and the formation of the metal EDTA complex is governed by pH. Hence pH of the solution is kept nearly constant by adding a basic buffer NH_4OH- NH_4Cl Buffer solution (pH~ 9-10)

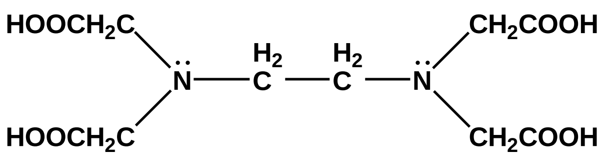
Estimation of hardness by EDTA is based on following principle:

1. The indicator EBT which is blue in color forms less stable wine-red colored complex with Ca^{2+} and Mg^{2+} Ions in hard water at pH of (9-10).
2. As this solution is titrated against EDTA the free Ca^{2+} and Mg^{2+} ions in water form stable metal-ion EDTA complex (colorless)
3. On addition of more amount of EDTA solution the metal ions from less stable {M-EBT} complex set free and form {M-EDTA} which is more stable. The solution turns blue due to free EBT.

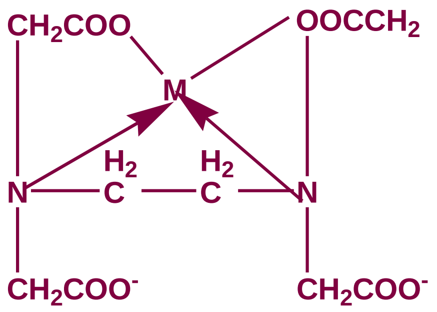
$$\mathbf{M^{2+}\ (M=Ca, Mg) + EBT \xrightarrow{pH\sim 9\text{-}10} \{M\text{-}EBT\}\ (less\ stable, Wine - red\ complex)}$$

$$\mathbf{M^{2+} + EDTA \xrightarrow{pH\sim 9\text{-}10} \{M\text{-}EDTA\}\ (More\ stable\ colourless\ complex)}$$

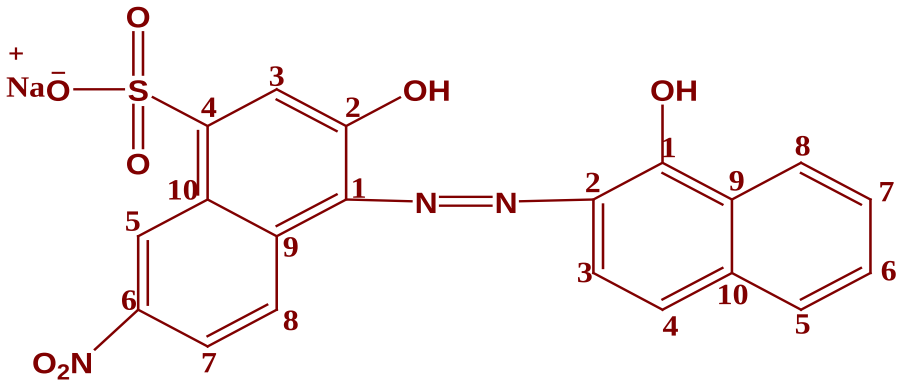
$$\mathbf{\{M\text{-}EBT\} + EDTA \xrightarrow{pH\sim 9\text{-}10} \{M\text{-}EDTA\} + EBT\ (Blue\ colour)}$$



Structure of EDTA



Structure of {M-EDTA} Complex



Structure of EBT

PROCEDURE: -

1. Prepare standard hard water by dissolving 1 g of $CaCO_3$ in one litre of distilled water.
2. Take 25 ml of standard hard water in to a conical flask.
3. Add 2 ml of buffer solution and 2-3 drops of EBT indicator to it.
4. The color of the solution becomes wine red, titrate it against standard EDTA solution till the color changes from wine red to blue at the end point.
5. Repeat the titration to get concordant values
6. Now take unknown hard water sample and repeat the same process

OBSERVATION TABLE: -

(i) Standardization of EDTA solution using standard hard water

Volume of standard hard water (SHW) taken for each titration = 25 ml.
Volume of 0.2N EDTA solution used = V_1 ml.

S.NO.	Volume of the solution taken in the titration flask	Burette readings		Volume of the titrant used
		Initial Reading	Final Readings	
1 2 3				

SHW EDTA

$N_1V_1 = N_2V_2$

$$0.02 \times 25 = N_2 \times V_1 \longrightarrow N_2 = \frac{0.02 \text{ X } 25}{V_1}$$

(ii) Determination of hardness of unknown hard water sample

Volume of unknown hard water (HW) taken for each titration = 25 ml.
Volume of 0.2N EDTA solution used = V_2 ml.

S.NO.	Volume of the solution taken in the titration flask	Burette readings		Volume of the titrant used
		Initial Reading	Final Readings	
1 2 3				

HW EDTA

$N_1V_1 = N_2V_2$ where $N_2 = \frac{0.02 \text{ X } 25}{V_1}$

$$N_1 \times 25 = N_2 \times V_2 \longrightarrow N_1 = \frac{0.02 \text{ X } 25}{V_1} \text{ X } \frac{V_2}{25} = 0.02 \text{ X } V_2/V_1$$

Strength = Normality X Eq.wt X 10^3 ppm (mg/lt)

RESULT: - Ca^{2+} and Mg^{2+} Hardness of unknown sample is____ppm.

PRECAUTIONS: -

1. Use distilled water for washing and rinsing of glass apparatus
2. Prepare EDTA solution in double distilled water.
3. Add same amount of indicator each time
4. Maintain pH from 9-10 during the titration by adding buffer.
5. Correctly observe the end point.

Guidelines for preparation of solutions:

Determinations of Ca^{2+} and Mg^{2+} hardness of water sample by using EDTA solution.

Preparation of 0.1M (0.2N) EDTA solution

Dissolve 37.225g of pure and dry EDTA disodium salt (M.W. 372.25) in distilled water and make up the volume to 1 litre.

Preparation of 0.01M (0.02N) $CaCO_3$ solution (SHW)

Weigh 1g of $CaCO_3$ (M.W. 100) and transfer it to one litre measuring flask. Add dil. HCl drop by drop till there is effervescence and the salt is completely dissolved. Add more distilled water to make up the volume to one litre.

Preparation of Ammonia buffer solution

Dissolve 70g of NH_4Cl in 570mL of Conc. NH_3 and dilute it to one litre with distilled water.

Preparation of EBT solution

Dissolve 0.5g of indicator in 100ml methanol.

Safety Instructions

1. NH_3 (Conc.):- It can cause serious burns to skin and eyes and its fumes irritate the eyes and respiratory system. Wear gloves, use the solution under hood and do not breathe its vapours.
2. HCl:- It is poisonous and corrosive. Contact or inhalation can cause severe damage to the eyes, skin and respiratory tract. Wear gloves and dispense under a hood, avoid contact and do not breathe the vapour.
3. CH_3OH:- It is flammable and harmful if ingested, inhaled or absorbed through skin. Avoid contact with the liquid and do not breathe its vapour.

Viva voce:

1. Define hardness of water?
2. What do you mean by temporary and permanent hardness?
3. What is the principle of EDTA titration?
4. How will you find out permanent hardness of water?
5. Why is it necessary to add buffer sol. to the water sample in determination of hardness by EDTA method?

EXPERIMENT:-2

AIM: - To determine the alkalinity of a given water sample.

CHEMICALS USED: - N/10 HCl, phenolphthalein and methyl orange indicators, sample water.

APPARATUS REQUIRED: - Burette, pipette, conical flask, beakers, funnel, and dropper

PRINCIPLE: - Alkalinity of water means the total content of those substances in it which causes an increased OH^- ion concentration up on dissociation or due to hydrolysis. The alkalinity of water is attributed to the presence of

(i) Caustic alkalinity(Due to OH^- and CO_3^{2-})
(ii) Temporary hardness (Due to HCO_3^-)

Alkalinity is a measure of ability of water to neutralize the acids.

Determination of alkalinity

OH^-, CO_3^{2-} and HCO_3^- can be estimated separately by titration against standard acid using phenolphthalein and methyl orange as indicators.

The determination is based on the following reactions

$$\left.\begin{array}{l} \left.\begin{array}{l} \text{(i) } OH^- + H^+ \longrightarrow H_2O \\ \text{(ii) } CO_3^{2-} + H+ \longrightarrow HCO_3^- \end{array}\right] P \\ \text{(iii) } HCO_3^- + H^+ \longrightarrow H_2O + CO2 \end{array}\right] M$$

The titration of water sample against a standard acid up to phenolphthalein end point (P) indicates the completion of reaction (i) and (ii) only. This amount of acid used thus corresponds to OH^- plus one half of the normal CO_3^{2-} present.

On the other hand, titration of the water sample against a standard acid to methyl orange end point (M) marks the completion of reaction (i), (ii) and (iii). Hence the total amount of acid used represents the total alkalinity. Thus,

$$P = OH^- + \tfrac{1}{2} CO_3^{2-}$$

$$M = OH^- + CO_3^{2-} + HCO_3^-$$

The **Table-1** below shows the type and amount of alkalinity in water

S.No	Result of Titration	OH^- ion	CO_3^{2-}	HCO_3^-
1	P=0	Nil	Nil	M
2	P=M	P or M	Nil	Nil
3	$P=1/2M(V_1=V_2)$	Nil	2P	Nil
4	$P>1/2M(V_1>V_2)$	2P-M	2(M-P)	Nil
5	$P<1/2M(V_1<V_2)$	Nil	2P	M-2P

PROCEDURE: -

1. Pipette out 20 ml of water sample into a conical flask. Add 1-2 drops of Phenolphthalein indicator.
2. Rinse and fill the burette with N/10 HCl.
3. Titrate the water sample in conical flask with N/10 HCl till the pink color just disappears.
4. Note down the reading and repeat to get concordant readings.
5. Again take 20 ml of water sample in conical flask and add methyl orange indicator to it.
6. Titrate the water sample in conical flask with N/10 HCl till the yellow orange color changes to orange red.
7. Note down the reading and repeat to get concordant readings.

OBSERVATION TABLEs: -

a) Using Phenolphthalein (Table-2)

Normality of the acid used: N/10

S.NO	Volume of the solution taken in the titration flask (in mL)	Burette readings		Volume of the titrant used
		Initial Reading	Final Readings	
1 2 3	20 20 20			Say V_1ml =P

b) Using Methyl orange (Table-3)

Normality of the acid used: N/10

S.NO.	Volume of the solution taken in the titration flask(in ml)	Burette readings		Volume of the titrant used
		Initial Reading	Final Readings	
1	20			Say V_2 ml = M
2	20			
3	20			

RESULT: - Alkalinity of the given water sample is__ ppm.

PRECAUTIONS: -

1. All glass wares should be properly washed.
2. Before use, rinse burette and pipette properly
3. Find the correct end point.

GENERAL CALCULATIONS: -

The volume required to neutralize particular ion can be calculated using **Table-1. For e.g.**

$P>1/2M$ i.e. $V_1>1/2V_2$

OH^- and CO_3^{2-} ions are present

From **Table-1**

Volume of aid required to neutralize $[OH^-] = 2P-M$ i.e. $\{2(V_1) - V_2\}$ml

Volume of aid required to neutralize $[CO_3^{2-}] = 2(M-P)$ i.e. $\{2(V_2-V_1)\}$ ml

Alkalinity due to OH^-

Acid Water

$N_1V_1 = N_2V_2$ where V_2=20mL

$1/10\{2(V_1)-V_2\} = N_2 \times 20$

$$N_2 = \frac{2(V_1)-V_2}{10 \times 20}$$

$$\text{Strength} = N2 \times \text{Eq. Wt of } CaCO_3 \text{ g/lt} = 2(V1)-V2 \times \frac{50 \times 10^3}{200} \text{ mg/lt or ppm}$$

In the same way the strength of other alkalinity causing ions can be calculated

Viva voce:

1. Write structural formula of phenolphthalein. In what forms does it exist in acidic and alkaline form.
2. What is alkalinity of water? How is it estimated?
3. Why OH- and HCO^{3-} ions cannot exist together in water?
4. What are the draw backs of using highly alkaline water?
5. What is caustic embrittlement and what are its drawbacks?
6. What are the disadvantages of scale formation?
7. A 200 ml of a water sample required 20 ml N/50 Sulphuric acid using methyl orange as indicator but did not give any coloration with phenolphthalein. What type of alkalinity is present? Express in mg/L.

Guidelines for preparation of solutions

1. **Methyl orange indicator:** Dissolve 20mg of methyl orange in 100 ml of hot water. Allow to cool and filter if necessary.
2. **Phenolphthalein indicator**: Dissolve 1g of Phenolphthalein in 100ml of 95% alcohol.
3. **0.1N HCl**: Measure 10mL of Conc. HCl (12N) in to one liter measuring flask. Pour 30mL distilled water and shake. Add more distilled water to make the solution up to the mark. Stopper the flask and shake vigorously.

Safety Instructions

HCl:- It is poisonous and corrosive. Contact or inhalation can cause severe damage to the eyes, skin and respiratory tract. Wear gloves and dispense under a hood, avoid contact and do not breathe the vapour.

EXPERIMENT:-3

AIM: **-** To determine the amount of dissolved oxygen in the given water sample by Winkler's method, a standard solution of $K_2Cr_2O_2$ of strength 0.01N is given.

CHEMICALS USED: - Sodium thiosulphate, (N/40), $MnSO_4$ KI, Starch, Conc. sulphuric acid

APPARATUS REQUIRED: - Burette, pipette, conical flask, beaker etc.

PRINCIPLE:- Oxygen itself is not a pollutant but its deficiency is an indicator of several types of pollution in water. Determination of dissolved oxygen (D.O.) is important for Industrial purposes. Dissolved Oxygen is needed for living organism to maintain their biological process. In presence of good amount of dissolved oxygen, aerobic bacteria lead to oxidation of organic compounds present in water. This kind of oxidation is called aerobic oxidation. But if the dissolved oxygen is less than **5ppm**, anaerobic oxidation of organic compounds present in water takes place by anaerobic bacteria. If the water is polluted with large amount of organic matter, a large amount of dissolved oxygen is rapidly consumed in the biological aerobic oxidation. This in turn decreases the population of aquatic life. D.O. is also important in precipitation and dissolution of inorganic substances in water. Dissolved oxygen is an important factor in corrosion.

Oxygen is poorly soluble in water. The solubility of oxygen decreases with increase in conc. of the salt under a pressure of one atmosphere, the solubility is less in saline water.

The estimation of dissolved oxygen in water is useful in studying corrosion effect of boiler feed water and in studying water pollution.

Dissolved oxygen is usually determined by Winkler's method. It is based on the fact that dissolved oxygen oxidized potassium iodide (KI) to iodine. The liberated iodine is titrated against standard sodium thio-sulphate solution using starch indicator. Since dissolved oxygen in water is in molecular state. It as such cannot oxidize KI. Hence Manganese Hydroxide is used as an oxygen carrier to bring about the reaction between KI and Oxygen. Manganese hydroxide, in turn, is obtained by the action of NaOH on $MnSO_4$.

$$MnSO_4 + 2NaOH \rightarrow Mn(OH)_2 + Na_2SO_4$$

$$2Mn(OH)_2 + O_2 \rightarrow 2MnO(OH)_2$$

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

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

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

$$\text{Starch} + I_2 \longrightarrow \text{Blue colored complex.}$$

The Nitrites present in water interfere with the titration as these can also liberate I_2 from KI.

$$2HNO_2 + H_2SO_4 + 2KI \rightarrow 2NO + K_2SO_4 + 2H_2O + I_2.$$

PROCEDURE:-

TITRATION – 1 STANDARDIZATION OF SODIUM THIOSULPHATE-

The burette is washed and rinsed with sodium thio-sulphate solution. Then the burette is filled with given sodium thio-sulphate solution. 20ml of 0.01N potassium dichromate solution is pipette out into a clean conical flask. To this 5ml of sulphuric acid and 15 ml of 5% potassium iodide are added. This is titrated against sodium thio-sulphate solution. When the solution become straw yellow colour, starch indicator is added and then the titration is continued . The end point is indicated by disappearance of blue colour and appearance of light yellow colour. The titration is repeated to get the concordant value.

TITRATION – 2 ESTIMATION OF DISSOLVED OXYGEN

250 ml of water sample is taken in an iodine flask, 2ml of manganese sulphate and 2ml of alkaline-iodide are added. The stopper is replaced and the flask is shaken several times for the rough mixing of reagents. The flask is left aside for some times. When half of the precipitate is settled down, the stopper is removed and 2ml of conc. sulphuric acid is added .The stopper is replaced and kept shaken for several times for complete dissolution of the precipitate. 100ml of this solution is pipette out and titrated against standard sodium thiosulphate solution. When the solution becomes light yellow starch indicator is added. The titration is repeated until the blue color is disappears. From the titer value the strength of dissolved oxygen is calculated and hence the amount of dissolved oxygen in the water sample is calculated.

RESULT:-

Amount of dissolved oxygen in tap water is = ………………………mg/lt.

Step-1

OBSERVATION TABLE:- STANDARDIZATION OF SODIUM THIOSULPHATE

TITRATION – 1

POTASSIUM DICHROMATE VS SODIUM THIOSULPHATE

S.NO.	Volume of potassium dichromate (ml)	Burette readings		Volume of sodium thiosulphate(ml)
		Initial Reading	Final Readings	
1				
2				
3				

Calculation:

Volume of potassium dichromate V_1 = 20ml

Strength of potassium dichromate N_1=0.01N

Volume of sodium thiosulphate V_2 =…………….ml

Strength of sodium thiosulphate N_2= ……?

$$V_1N_1=V_2N_2$$
$$N_2=V_1N_1/V_2$$

Strength of sodium thiosulphate = N_2= 20×0.01/V_2
=……………..
=……………….

Step 2:- Estimation of dissolved oxygen

Titration – 2 Water sample v/s sodium thio-sulphate

S.NO.	Volume of the solution taken in the titration flask	Burette readings		Volume of the titrant used
		Initial Reading	Final Readings	
1	100			
2	100			
3	100			

Calculations:
Volume of sodium thiosulphate V_1 = …………………. ml
Strength of sodium thiosulphate N_1 = ………………… N
Volume of water sample V_2 = 100ml
Strength of dissolved water sample N_2 =?

$$V_1N_1=V_2N_2$$
$$N_2= V_1N_1/V_2$$

Amount of dissolved oxygen in One litre of tap water = normality × eq.wt.of O_2×1000
= ……………….N_2×8×1000

RESULT: - Dissolve oxygen in the sol. is____ mg/l or ppm.

PRECAUTIONS: - 1. Accurate reading should be taken.
2. The indicator to be added should be adjusted in a manner to give accurate end point.
3. Be careful while sucking KI sol. as it is highly poisonous.

Viva voce:

1. Why $MnSO_4$ is used in above determination?
2. Why azide is added to the reaction mixture?
3. What is the significance of above determination?
4. What is the end point if we use methyl orange as indicator in titration?

EXPERIMENT: - 4

AIM:- To find the eutectic point for a two component system by using method of cooling curve.

CHEMICALS USED: - Urea, phenol, Colorless liquid (Paraffin/glycerin)

APPARATUS REQUIRED: - Boiling tube, test tubes, thermometer 360^0, Stirrer, beaker, stop watch.

PRINCIPLE:-

Eutectic System:- A binary system consisting of two substances, which are miscible in all proportions in the liquid phase, but do not react chemically is known as “Eutectic (Easy to melt) system”, e.g. A mixture of lead and silver comprises of such a system.

Eutectic Mixture is a solid solution of two or more substances having the lowest freezing point of all the possible mixture of the components. Example: Alloys of low melting point

Eutectic Point:- Two or more solid substances capable of forming solid solutions with each other, have the property of lowering each other’s freezing point and the minimum freezing point attained, corresponding to the Eutectic mixture, is termed as Eutectic Point (Mean lowest M.P)

Applications of Eutectics

Low melting alloys are used in safety devices (e.g. as plugs in automobiles), fire sprinklers and as fail-safe device in boilers. By suitable choice of metals, very low melting alloys can be obtained. e.g. wood’s metal (Alloy containing 50% Bi, 25% Pb, 12.5%, Cd, 12.5% Sn) melts at 65° C only.

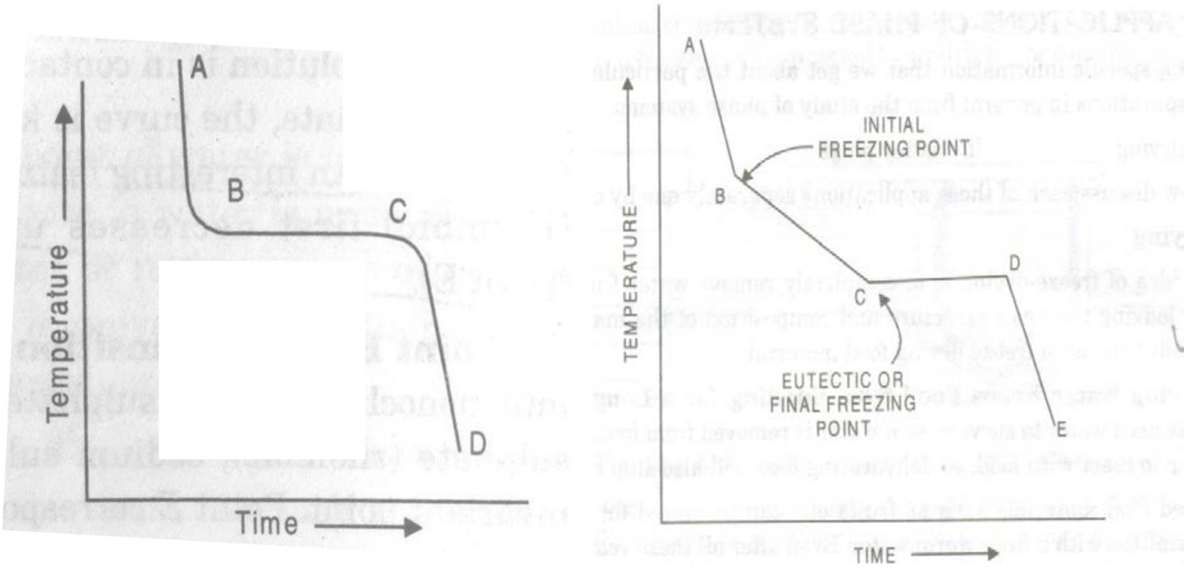
Congruent melting compound:-

The melting point of phenol is 43° C and that of urea is 132° C. When they are heated together, they chemically react with each other to form a thermally stable compound which is called congruent melting compound. The congruent melting compound has melting point of 61° C and its composition is **Urea: phenol = 1:2**

In thermal analysis curve, congruent melting point is observed as hault i.e. when temperature becomes constant

The purpose of this experiment is to obtain data by thermal analysis for constructing a phase diagram which indicates the solid and liquid phase that are present at each temperature and composition. The method consists of cooling of a molten mixture of the components and a graph, known as cooling curve, between temperature and time is drawn. **Cooling curve** is continuous curve as long as there is no phase change during the cooling. When the solid starts separating, the rate of cooling is decreased due to liberation of heat.

In order to obtain a phase diagram, mixture of two compounds is prepared. This mixture is heated to a liquid state and then cooling curve is obtained for the mixture.



(a) (b)

Diagram:-Cooling Curves of (a) Pure substance (b) Mixture of two components

PROCEDURE:

1. Prepare a mixture of 2.5g urea and 5g phenol
2. Melt the mixture by placing the test tube in the bath of hot liquid paraffin or glycerin
3. When the whole mass has melted, remove the tube from the bath, wipe it clean and fit it with cork and thermometer. The tube is then fitted in boiling tube which serves as a air jacket.
4. Start the stop watch and note the temperature at different intervals of time (say after every min)
5. Allow the temperature to fall still further
6. (i.e., allow the cooling to continue) and find out the temperature at which the solid and liquid systems show temperature halts before complete solidification. Lowest temperature halt will be the Eutectic point.

OBSERVATION TABLE

Mixture (Composition: Urea + Phenol = 1:2)

Time in minutes	Temperature in ° C
-------------	……….
------------	……….
------------	……….

Graph:- Draw a cooling curve by plotting time of cooling in minutes along the X-axis and temperature in ° C along Y-axis. From the cooling curve find the eutectic temperature of the mixture.

RESULT:- The Eutectic point of Urea-phenol system experimentally found is ---------------° C

PRECAUTIONS:-

1. Weigh the quantity of urea and phenol accurately to get exact Eutectic mixture
2. Read the temperature carefully and accurately
3. Handle the phenol carefully.

Viva voce:

1. Define the following terms:
   Eutectic Point, Eutectic Mixture, Congruent M.P, Congruent melting compound, Phases, Components, Degree of Freedom
2. Give few applications of Eutectic Mixture.
3. Explain "Eutectic is a mixture not a compound"
4. What is the purpose of using boiling tube in this experiment?

Safety Instructions:

Phenol:- Most phenols are harmful if inhaled, ingested or absorbed through skin. They cause severe irritation or damage to skin and eyes. Some phenols are suspected carcinogen, should not inhale its dust or vapor, wear gloves and avoid contact.

EXPERIMENT: - 5

AIM: - Determination of viscosity of lubricant by Red Wood Viscometer (No. 1 & No. 2)

CHEMICALS USED: - Given sample of lubricant, suitable organic solvent like CCl_4, ether, petroleum spirit or benzene.

APPARATUS REQUIRED: - Red Wood viscometer no. 1 & no. 2, stop watch, Kohlrausch flask, thermometer, filter paper

PRINCIPLE: -

Viscosity is the property of a fluid that determines its resistance to flow. It is an indicator of flow ability of a lubricating oil; the lowest the viscosity, greater the flow ability. It is mainly due to the forces of cohesion between the molecules of lubricating oil.

Absolute Viscosity may be defined as “the tangential force per unit area which is required to maintain a unit velocity gradient between two parallel layers. It is denoted by η(eta). Its Unit in CGS system is poise and its dimensions are $ML^{-1}T^{-1}$.

Viscosity Index: Viscosity generally decreases with increase in temperature. The maintenance of viscosity over the range of temperature is called the viscosity Index (V.I)
A relatively small change/no change in viscosity with temperature is indicated by high viscosity index whereas low viscosity index shows relatively large change in viscosity with temperature

Note: There is a direct correlation between molecular structure of lubricating oil with its viscosity and viscosity index. A high viscosity index is exhibited by those lubricating oils which have linear or rod like shape molecules with high molecular weight. This is due to the greater intermolecular forces of attraction

Effect of temperature on viscosity

Viscosity of lubricating oil is inversely proportional to the temperature i.e. with increase of temperature, viscosity decreases. This is due to the decrease in intermolecular attraction
At higher temperature, oil must have sufficient viscosity to carry loads. Hence heavier oils are used at higher temperature. Similarly, light oils are used at low ambient temperature

Effect of pressure on viscosity

Lubricating oils are subjected to extreme pressure at the interphase between gears and rolling element. At such higher pressure, viscosity of lubricating oil increases considerably.
Vicosity helps in selecting good lubricating oil.

Light oils	**Heavy Oils**
Having low density	High density
Easy flow ability	Low flow ability
Used for; High speed, low pressure & Low temperature	Used for; Low speed, high pressure & high temperature

Significance of viscosity measurements

Viscosity is the property of lubricating oil that determines it ability to lubricate and through its film strength, viscosity values are used

- In evaluating load carrying capacity
- In denoting the effect of temperature changes and for determining the presence of contaminants in used oil during service.
- Absolute viscosity values are required for use in all bearing design calculations and other lubrication engineering technical design problems.

Measurement of viscosity of lubricating oil

The instruments used for measuring the viscosity are known as viscometers

Different types of viscometers are

- Saybolt Viscometer
- Angler's Viscometer
- Ostwald Viscometer
- Kinematic Viscometer
- Redwood Viscometer

Redwood Viscometer

It is of two types

a) Redwood viscometer No.1- Universal
b) Redwood viscometer No.2- Abmiralty

Both the above viscometers are identical in principle, shape and mode of testing. The essential differences between the two are

	Redwood viscometer No.1- Universal	Redwood viscometer No.2- Abmiralty
Dimensions of orifice	Length-10mm, Dia-1,62mm	Length-50mm, Dia-3.8mm
Kohlrausch flask	Smaller mouth	Wider mouth
Useful for	Low viscous oil having flow time between 30s-2000s e.g. Kerosine oil and mustard oil	Higher viscous oils having flow time greater than 2000s e.g. Fuel oil, mobile oil

Description of the Redwood viscometer

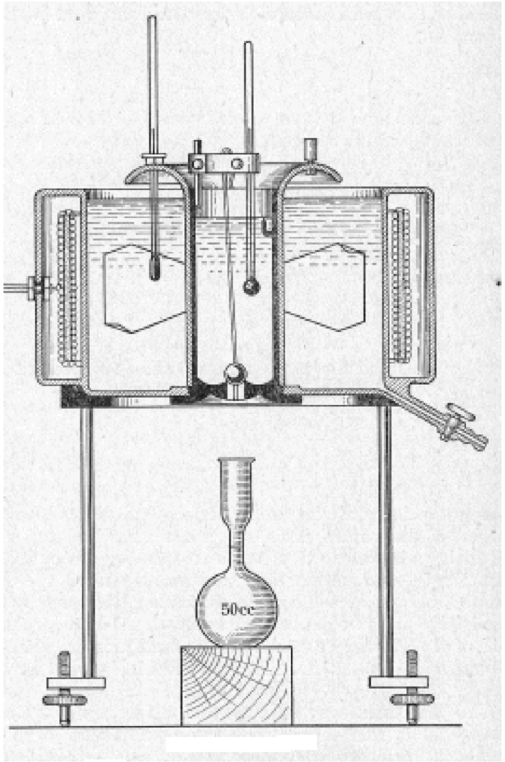
It is divided in to three parts

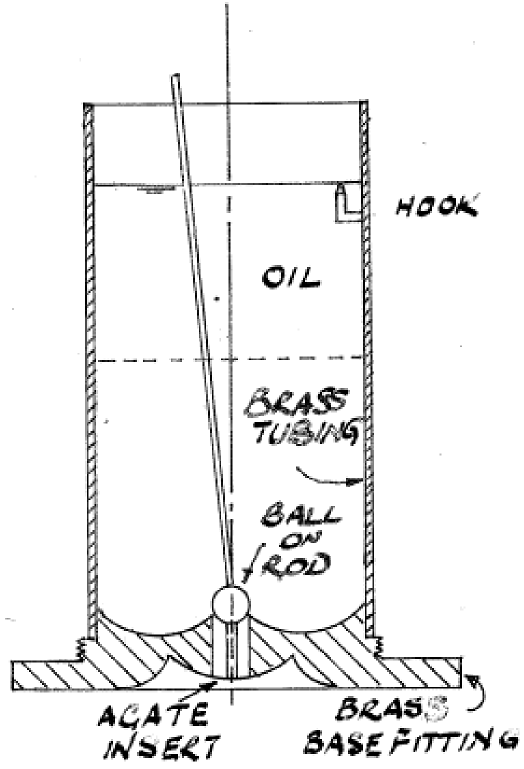
1. **Oil Cup**;
   Material- Silver plated brass
   Height-90mm
   Diamtere-46.5mm

It holds the test sample of lubricating oil. The bottom of the cup is fitted with polished-agate discharge tube containing an orifice of specified dimension

2. **Water Bath**
   Oil cup is surrounded by water bath for adjusting the temperature

3. **Kohlrausch Flask**
   It receives the oil from polished-agate discharge tube





PROCEDURE: -

1. Select the appropriate viscometer, either Redwood viscometer No.1 or 2 depending up on the nature of lubricating oil
2. Clean the viscometer cup properly with the help of suitable solvent e.g. CCl_4, ether, petroleum spirit or benzene and dry it to remove any traces of solvent.
3. Level the viscometer with the help of leveling screws.
4. Fill the outer bath with water for determining the viscosity at 80 ^{o} c and below.
5. Place the ball valve on the jet to close it and pour the test oil into the cup up to the tip of indicator.
6. Place a clean dry Kohlrausch flask immediately below and directly in line with discharging jet.
7. Insert a clean thermometer and a stirrer in the cup and cover it with a lid.
8. Heat the water filled in the bath slowly with constant stirring. When the oil in the cup attains a desired temperature, stop the heating.
9. Lift the ball valve and start the stop watch. Oil from the jet flows into the flask.
10. Stop the stop watch when lower meniscus of the oil reaches the 50 ml mark on the neck of receiving flask.
11. Record the time taken for 50 ml of the oil to collect in the flask.

12. Repeat the experiment to get more readings.

S.NO.	Temperature	Viscosity, t (seconds, Redwood No. 1)
1 2 3		Mean value:

OBSERVATION TABLE:

RESULT: - The viscosity of given oil sample using Redwood viscometer no. -------- at___ ^{o}C is ____ Red Wood seconds.

PRECAUTIONS: -

1. The oil should be filtered thoroughly a muslin cloth to remove solid particles that may clog the jet.
2. The receiving flask should be placed in such a manner that the oil stream from jet strikes the neck of receiving flask and do not cause any foaming.
3. After each reading the oil should be completely drained out of receiving flask.

Viva voce:

1. What is viscosity? Define Absolute viscosity
2. How does the viscosity of liquid vary with temperature?
3. What is viscosity index? How viscosity index is determined?
4. Name few lubricants with high and low viscosity index.
5. What is the difference between Red wood viscometer No.1 and 2?
6. How viscosity index of lubricating oil is can be improved?

EXPERIMENT: -6

AIM: - To determine flash point and fire point of the given lubricating oil using Pensky Marten's apparatus.

CHEMICALS USED: - Given lubricating oil, suitable organic solvent like CCl_4, ether, petroleum spirit or benzene

APPARATUS REQUIRED: - Pensky Marten's flash and fire point apparatus, match box, thermometer, and filter paper.

PRINCIPLE: -

Flash point is the lowest temperature at which the lubricating oil gives off enough vapors that ignite for a moment when tiny flame is brought near it.

Fire point is the lowest temperature at which the vapors of the oil burn continuously for at least five seconds when a tiny flame is brought near it.

Significance

Flash and fire points are used to indicate

- Fire hazard of petroleum products and evaporation loses under high temperature loses
- It gives us the idea about the maximum temperature below which the oil can be used
- It is used as the means of identification of specific lubricating oil
- For detection of contamination in the given lubricating oil

Description of Pensky Marten's apparatus

It is used to determine the flash point of the lubricating oils, fuel oils, solvents, solvent containing material and suspension of solids.

It consists of three parts

a) **Oil Cup**

Material- Brass
Height – 5.5cm
Diameter-5cm

Lid of the cup is provided with four openings of standard sizes, first opening is for stirrer, second is for admission of air, third is for thermometer and fourth is for introducing test flame.

b) **Shutter**

At the top of the cup shutter is provided. By moving the shutter, opening in the lid opens and flame is dipped in to this opening, bringing the flame over the oil surface. As the test flame is introduced in the opening, it get extinguished, but when the test flame is returned to its original position, it is automatically lightened by the pilot burner

c) Stove

It consists of 1. Air bath, 2. Top plate on which the flange of the cup rest

Pensky Marten’s (closed cup) flash point apparatus

PROCEDURE: -

1. Clean and dry all parts of the apparatus with the help of suitable solvent e.g. CCl_4, ether, petroleum spirit or benzene and dry it to remove any traces of solvent.
2. Fill the oil cup with the test oil up to the mark.
3. Fix the lids on the top through which are inserted a thermometer and a stirrer. Ensure that the flame exposure device is fixed on the top.
4. Light the test flame and adjust it to about 4 mm in diameter.
5. Heat apparatus as temperature of oil increases by 5 to 6^0 per min. as stirrer is continuously rotated.
6. At every 1^0 C rise of temp. Introduce test flame into the oil vapor. This is done by operating the shutter. On moving knob of shutter, test flame is lowered in oil vapors through opening.
7. When test flame causes a distinct flame in interior cup, note temp. Which represent the flash point?
8. Further heat the oil at the rate of 1^0C/ min. and continue applying the test flame as before.
9. The temperature at which the vapors of the oil give a clear and distinct blue flash for five seconds is recorded as the fire point of the oil.

RESULT: -

The flash point of given oil sample =___˚C.

The fire point of given oil sample =___˚C.

PRECAUTIONS: -

1. The apparatus should be thoroughly dried. There should be no trace of moisture inside the cup.
2. The thermometer bulb should dip into the oil.

3. While applying the test flame, stirring should be continued.
4. Fill the sample of the lubricating oil up to the mark. There should be no oil on the outer part of the cup.
5. Avoid breathing over the surface of the oil.

Viva voce:
1. Define flash point and fire point of lubricating oil.
2. What should be the flash point of a good lubricant?
3. In Pensky Martin’s apparatus what is the purpose of air jacket surrounding the oil cup.
4. What are the factors affecting the flash point and fire point?

EXPERIMENT- 7(A)

AIM: - To prepare phenol formaldehyde resin (Bakelite).

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

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

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

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

Properties:-

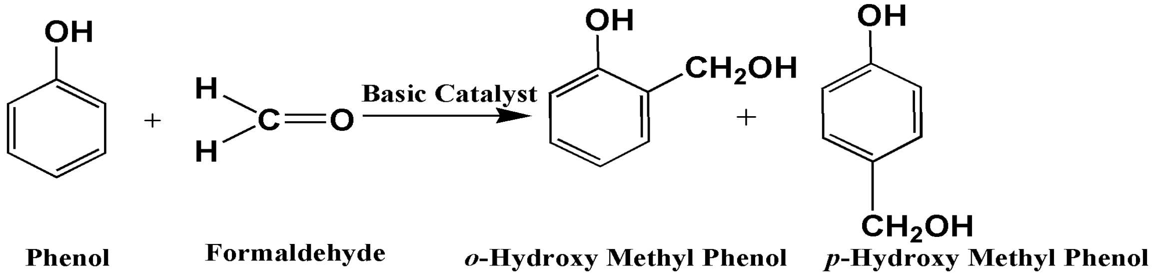
- Phenol- formaldehyde resins having low degree of polymerization are soft. They possess excellent adhesive properties and are usually used as bonding glue for laminated wooden planks and in varnishes and lacquers.
- Phenol- formaldehyde resins having high degree of polymerization are hard, rigid, scratch-resistant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

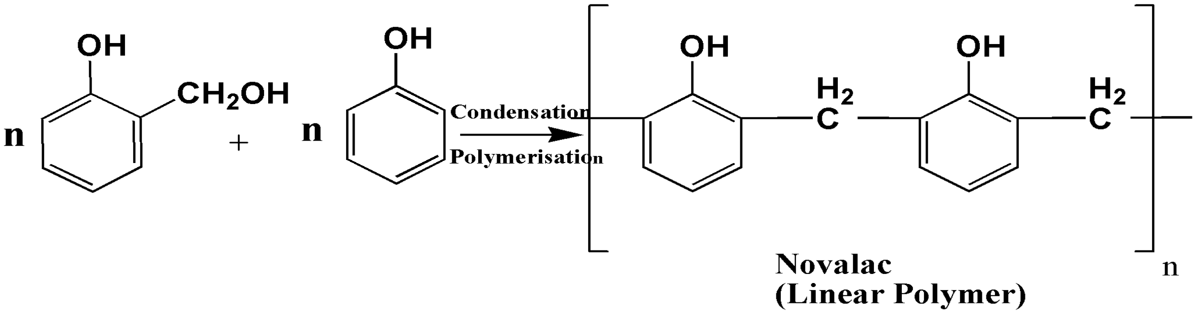
Uses:-

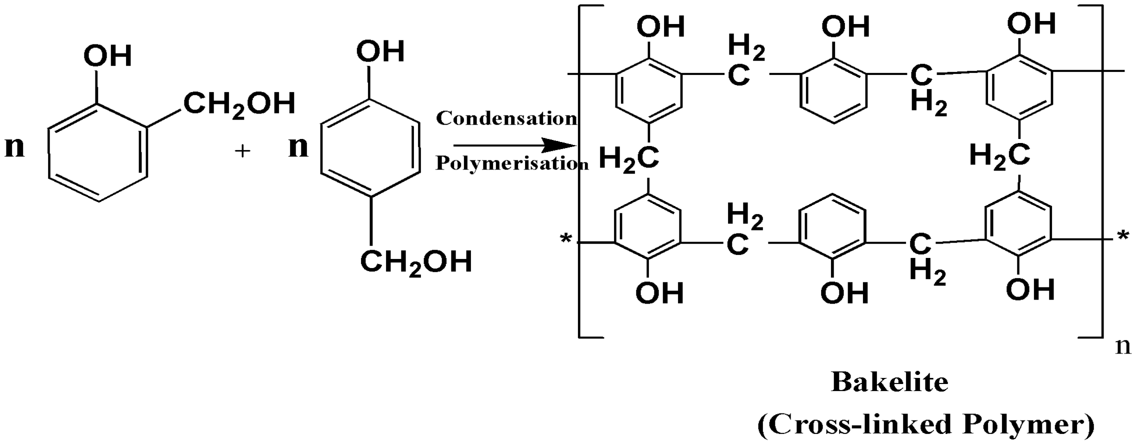
- They are used for making moulded articles such as radio and TV parts, combs, fountain pen barrels, phonograph records etc.
- They are used for making decorative laminates, wall coverings etc.
- They are used for making electrical goods such as switches, plugs etc.
- They are used for impregnating fabrics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

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

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







PROCEDURE: -

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

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

PRECAUTIONS: -

1. The reaction is sometimes vigorous and it is better to be a few feet away from the beaker while adding the H_2SO_4 and until the reaction is complete.
2. The experiment should be preferably carried out in fume cupboard.

Viva voce:

1. Phenol formaldehyde is also called as.
2. What do you understand by resin?
3. Give main uses of the phenol formaldehyde resin.
4. What type of co-polymer is phenol formaldehyde resin?
5. Briefly describe the properties of phenolic resins.
6. Write chemical equations for preparation of phenolic resins.

Safety Instructions:

1. Phenol:- Most phenols are harmful if inhaled, ingested or absorbed through skin. They cause severe irritation or damage to skin and eyes. Some phenols are suspected carcinogen, should not inhale its dust or vapor, wear gloves and avoid contact.
2. H_2SO_4:- It is poisonous and corrosive. Contact or inhalation can cause severe damage to the eyes, skin and respiratory tract. Wear gloves and dispense under a hood, avoid contact and do not breathe the vapour.

EXPERIMENT: - 7(B)

AIM: - To prepare Urea-formaldehyde resin.

CHEMICALS USED: - 40% formaldehyde, Urea, Conc. sulphuric acid, distilled water

APPARATUS REQUIRED: - Beakers, glass rod, funnel, measuring cylinder, filter paper.

PRINCIPLE: - Urea formaldehyde resin is prepared by the condensation reaction between urea and formaldehyde in neutral or acidic conditions.

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

Properties;-

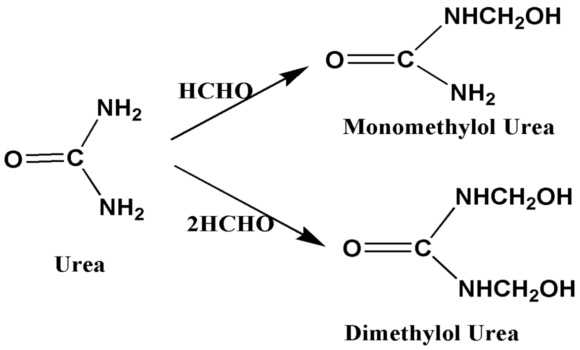
- They are clear and white.
- Better hardness and tensile strength than PF resins.
- Resistant to most of the solvents and grease.
- Excellent abrasion resistant and stable to light.
- Good adhesive characteristics.
- Good electrical insulators and possess chemical resistance.
- They can be synthesized in any desired colour by adding proper pigment and filler during synthesis

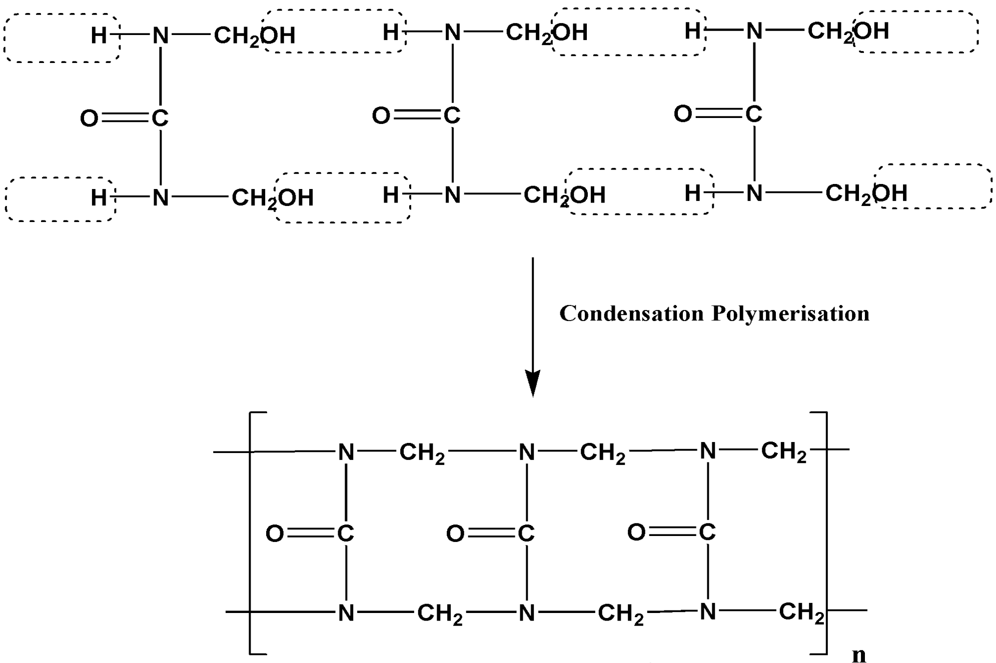
Uses:-

- These resins are widely used in manufacture of buttons, bottle caps, house hold appliances, surgical items etc.
- They are used as adhesives in plywood industries.
- They are also used in the manufacturing of enamels and other surface coatings.
- Used for the finishing of cotton textiles (They impart stiffness, crease resistance, fire retardation, water repellency. They are also helpful for shrinkage control)

Preparation:-

Urea and formaldehyde react with each other in neutral or acidic conditions to give mono and dimethylol urea, which undergo further condensation reaction to give linear, partially cross-linked or fully cross-linked polymer.





Urea-Formaldehyde Resin
Cross-linked Polymer

PROCEDURE: -

1. Place about 5ml of 40% formaldehyde solution in 100 ml beaker.
2. Add about 2.5 g of urea with constant stirring till saturated solution is obtained.
3. Add a few drops of conc. Sulphuric acid with constant stirring.
4. A voluminous white solid mass appears in the beaker.
5. Wash the white solid with water and dry it in the folds of filter paper.
6. Calculate the yield of the product

RESULT: - The yield of urea formaldehyde resin is ____ g.

PRECAUTIONS:-

1. Sulphuric acid should be added drop by drop with constant stirring.
2. White solid mass should be removed immediately as it sticks to the walls of the beaker.
3. The reaction mixture should be stirred continuously.
4. The reaction is sometimes vigorous and it is better to be a few feet away from the beaker while adding the H_2SO_4 and until the reaction is complete.

Viva voce:

1. What do you understand by resin?
2. Urea formaldehyde resin is an addition polymer or condensation polymer.
3. What are the main uses of the urea formaldehyde resin.
4. Briefly describe the properties of Urea formaldehyde resin.
5. Write chemical equations for preparation of Urea formaldehyde resin.

Safety Instructions

H_2SO_4:- It is poisonous and corrosive. Contact or inhalation can cause severe damage to the eyes, skin and respiratory tract. Wear gloves and dispense under a hood, avoid contact and do not breathe the vapour.

Points to be noted

1. H. Staudinger was awarded in 1953, a noble price of chemistry for the discovery of macro molecules.
2. Karl Ziegler and G. Natta were awarded in 1963, noble price for chemistry and technology of high polymers.
3. Pierre-Guilles Dgennes was awarded in 1991, physics noble price for contributing on methods to study liquid crystals and polymers.
4. A. J. Heeger, A.G. Macdiarmid and H. Shirakawa were awarded in 2000, chemistry noble price for the discovery and development of conducting polymers.

EXPERIMENT: -8

AIM: - Determination of saponification value of an oil.

CHEMICALS USED: - Standard N/2 HCl, Alc. NaOH and phenolphthalein.

APPARATUS REQUIRED: - Round bottom flask, burette, pipette, water condenser, water bath.

PRINCIPLE: - The saponification value of oil is the number of milligrams of KOH required to saponify 1 g of oil or fat. The vegetable or animal oil react with KOH to form the potassium salt of fatty acids. The saponification value of oil is determined by refluxing a known quantity of the sample with the known excess of a standard KOH sol. and determining the alkali consumed by titrating the unreacted alkali.

OBSERVATION:-

Sr.No.	Vol.of KOH (ml)	Vol.of HCl in burette		Total vol.of HCl (ml)
		initial	final	
1	25			
2	25			
3	25			

GENERAL CALCULATIONS: -

Weight of the empty beaker = w_1 g
Weight of the beaker and oil = w_2 g
Weight of the sample taken = $w_2 - w_1$
Volume of alcoholic KOH added to both flasks = 50 ml
Vol. of N/2 HCl used in sample determination = V_1 ml
Vol. of N/2 HCl used in blank determination = V_2 ml
Vol. of N/2 HCl equivalent to KOH used in saponify (w_2- w_1) g of oil. = V_2-V_1
mg of KOH present in 1000 ml of N/2 KOH = ½ * 56g = 28g = 28 * 1000mg.
mg of KOH present in 1 ml of N/2 KOH = 28/1000 * 1000 = 28mg.
mg of KOH present in V_2-V_1 ml of $N/_2$ KOH = V_2-V_1 * 28 mg
Saponification value of sample = (V_2-V_1) * 28/Amount of oil.

PROCEDURE: -

1. Weigh accurately 2 gm of oil sample into 250 ml flask.
2. Add 25 ml of N/2 alc. KOH into it. Add the same amount of solvent and N/2 alcoholic KOH to another flask for blank titration.
3. Fit the first flask with the water condenser and reflux the contents on water bath for half an hour.
4. Cool the contents lightly and disconnect the condenser correctly and rinse it with small amount of distilled water into flask.
5. Add two drops of phenolphthalein indicator and titrate the contents of the flask with standard solution of HCl (N/2) until the pink color just disappears.

RESULT: - The saponification value of given oil is____gm.

PRECAUTIONS: -

1. Distilled water should be used.
2. Drop of indicator should be adjusted in a manner to give accurate end point.

Viva voce:

1. What is saponification value?
2. Why mineral oils are not saponified?
3. What is the significance of determination of saponification no.?

EXPERIMENT: -9

Aim: To determine TDS of water samples of different sources.

Apparatus: China dish, Muffle furnace, hot air oven, balance, filter paper, Imhoffcone, conical flask etc.

Theory: The environmental engineer needs the data regarding the amount of solid matter in a wide variety of liquids ranging from portable water samples to polluted water samples. The solids refer to the matter that remains as residue upon evaporation and drying at 103-105 ^{o}C. The total solid may be either dissolved or suspended. The determination of solid content is very important for sewage/waste water samples because the process of treatment operation to be applied depends more or less on nature and quantity of solids present in waste water. The solid content may be volatile (organic) and fixed (inorganic) in nature.

Classification of Sewage Solids:

Total solids: Analytically the total solids content of a wastewater is defined as all the matter that remains as residue on evaporation at 103^{o}C and subsequent drying. These solids are very important as they indicate the strength of sewage and amount of treatment required.

Total solids in waste water exists in 3 forms

a. Dissolved solids
b. Colloidal solids
c. Suspended solids

Dissolved solids are those which remain dissolved in sewage just as salt in water and are generally organic in nature causing putrefaction and thus increasing the strength of sewage and creating nuisance if disposed off untreated.

Colloidal solids are finely divided solids remaining either in solution or in suspension.

Suspended solids are those, which are in suspension and are floating in sewage. They are further classified into:

1. Settleable solids, which settle down at the bottom of Imhoffcone and measure of the quality of sludge that will be removed by sedimentation.
2. Non settleable solids are those, colloidal in nature and can be removed by Chemical treatment.

Fixed solids: Fixed solids are also called inorganic solids consisting of minerals and salts, which include sand, gravel, debris, dissolved salts etc. They constitute about 55% of the total solids and are not harmful in sewage treatment operations and require only mechanical appliances such as screens for their removal.

Volatile solids:

They are also called organic solids or ignited solids, consisting of organic matter such as carbohydrates, fats, oils etc and constitute about 45% of the total solids and commonly used in the analysis of wastewater sludge's to measure their biological stability.

Procedure:

1. Total solids: Take 10 ml of well mixed sewage sample in a china dish and evaporate all the liquid by heating at a temperature of 103 ^{o}C for 1 to 1.5 hours in a hot air oven and weigh the residue which gives the amount of total solids in sewage.
2. Dissolved solids: Take 10 ml of well mixed sewage sample and filter it through Whatmann filter paper (No 44). Heat the filtrate at 103 ^{o}C for 1 to 1.5 hours in a hot air oven and weigh the residue, which gives the amount of Dissolved solids in sewage.
3. Suspended solids: The difference between the total solids and dissolved solids gives the amount of suspended solids.
4. Fixed solids: The china dish with the residue of total solids test is heated in a muffle furnace for 15 to 20 minutes at 600 ^{o}C. Cool the dish and take the weight of the residue with china dish, which gives the total fixed solids.
5. Volatile solids: The difference between the total solids and fixed solids gives the amount of volatile solids.
6. Settle able Solids: One litre of well-mixed sewage sample is poured into an Imhoff cone (1lt capacity, graduated upto 40ml) and allowed to stand for 45 minutes. Then stir the sides of the cone gently with a glass rod or spin the cone a few times to ensure that all solids adhering to the sides are loosened. Allow to settle for further 15 minutes and note down the reading on the cone which gives the total settleable solids in ml/lt

Calculations:

1. **TotalSolids**:
   Wt of clean empty china dish, W_1= .... gm
   Wt of china dish + residue of sewage sample oven dried at 103^{o}C, W_2 ....gm
   Total solids = [(W_2 - W_1) x 10^6] / ml of sample, mg/lt

2. **Dissolved Solids**:
   Wt of clean empty china dish, W_3= .... gm
   Wt of china dish + residue of filtered sewage sample
   (Through Whatmann filter paper No 44) oven dried at 103^{o}C, W_4..................... gm
   Dissolved solids = [(W_4 – W_3) x 10^6] / ml of sample, mg/lt.

3. **Suspended Solids**:
   Suspended solids = Total solids - Dissolved solids

4. **Fixed solids (In organic solids)**:
   Wt of clean empty china dish, W_5=... gm
   Wt of china dish + residue of Total solids test heated to 600 °C for 20 minutes in a muffle furnace, W_6 ....gm
   Fixed solids = $[(W_6 - W_5) \times 10^6]$ / ml of sample, mg/lt.

5. **Volatile Solids (Organic Solids)**: Volatile solids = Total solids – fixed solids

6. **Settleable Solids**: Settleable solids (from Imhoff cone), ml/lt.

Results:

Conclusion:

EXPERIMENT: -10

AIM: - Verify Beer's law and apply it to find the conc. of given unknown $KMnO_4$ solution spectrophotometrically.

CHEMICALS USED: - Distilled water, standard solutions of $KMnO_4$, tissue paper

APPARATUS REQUIRED: - UV- visible spectrophotometer, tissue paper, and beaker.

PRINCIPLE: - When an electromagnetic radiation is passed through a sample, certain characteristic wavelength is absorbed by the sample. As a result intensity of transmitted light is decreased. The measurement of the decrease in intensity of radiation is basis of spectrophotometry. According to the Beer Lambert's law the intensity of incident light is proportional to the length of thickness of the absorbing medium and the conc. of the solution.

PROCEDURE: - Setting of the instrument

1. Spectrophotometer should initially read zero on transmittance scale (T). If it doesn't read zero, set it mechanically with adjusting knob.
2. Connect the instrument to the mains and put on the power switch.
3. Adjust the wavelength knob to the required wavelength region on scale.
4. Choose the position of wavelength switch corresponding either to 340-400 or 400-960.
5. Adjust the meter needle on zero on transmittance scale and 100 on O.D. scale

Workin

6. Open the lid the cell compartment and insert a cuvette containing the black solvent (water). Close the lid/
7. Adjust the needle to 100% transmittance or zero optical density
8. Remove the cuvette and close the lid tightly again. Fill it with standard sol.
9. Now place the cuvette containing the standard sol. in the cell compartment. Note the O.D and transmittance.

Determination of λ max

10. Now change the wavelength by 20 nm and note absorbance
11. Plot a graph between wavelength on the X axis and absorbance on Y-axis.

Verification of Beer's law

12. Fix the wavelength at λ max position.

13. Prepare potassium per magnate sol. with conc. 0.2%, 0.5%, 1.0%, 1.5%, 2.0%, 2.5%, and 3.0%.
14. Note down absorbance of series;
15. Now find out OD of unknown sol. of $KMnO_4$.

OBSERVATION TABLE: -

Determination of λmax: -

Wavelength (nm)	Absorbance (OD)
1 2 3 4	

OBSERVATION TABLE: -

Verification of Beer's law: -

Concentration moles/lit.	Absorbance (OD)
1 2 3 4	

RESULT: - λ max. For $KMnO_4$. -------- _ nm.

PRECAUTIONS: -

1. Always use dilute sol. for getting calibration curve.
2. Cuvette should be cleaned properly and wiped away with tissue paper.

Viva voce:
1. What is spectrophotometry?
2. Discuss Beer's law of absorbance.
3. What is calorimetry?
4. How can you verify Beer's law?

EXPERIMENT-11

AIM:- Determination of strength of HCl solution by titrating against NaOH solution conductometerically

Principle :-

Solution of electrolytes conducts electricity due to the presence of ions. The specific conductance of a solution is proportional to the concentration of ions in it. The reaction between HCl and NaOH may be represented as,

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

When a solution of hydrochloric acid is titrated with NaOH, the fast moving hydrogen ions are progressively replaced by slow moving sodium ions.

As a result conductance of the solution decreases. This decrease in conductance will take place as there is an excess of hydroxide ions.

A graph is drawn between volume of NaOH added and the conductance of solution. The exact end point is intersection of the two curves.

MATERIAL REQUIRED

- Conductivity bridge
- Conductivity cell
- Beaker
- Standard N/10 HCl
- Given NaOH solution
- Burette, pipette, glass rod etc.,

PROCEDURE:-

The burette is filled with sodium hydroxide solution upto the zero level. 20 ml of the standard HCL is pipette out into a clean 100 ml beaker. The conductivity cell is placed in it and then diluted to 50 ml by adding conductivity water, so that the electrodes are well immersed in the solution. The two terminals of the cell are connected with a conductivity bridge. Now 1 ml of NaOH from the burette is added to the solution, takes in the beaker, stirred for some time and then conductivity is measured. (The conductivity is going on decreasing upto the end point). This process is repeated until at least five readings are taken after the end point has been reached.

Now the graph is plotted by taking volume of NaOH in the X-axis and conductance in the Y-axis. The end point is noted from the graph and the amount of NaOH present in 250 ml of the solution is calculated.

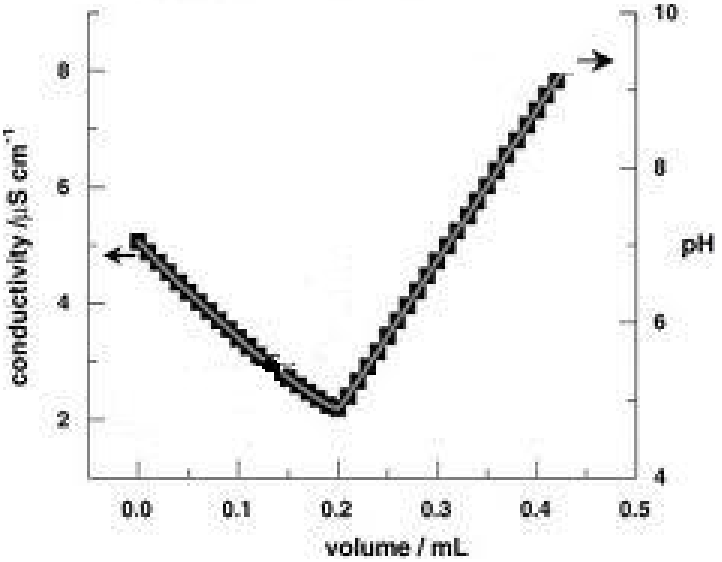


TABLE – I

Titration of standard HCl vs NaOH
Volume of HCl taken = 20 ml

Sr.No.	Vol.of NaOH(ml)	Conductance(mho)
1	0	
2	1.0	
3	2.0	

RESULT:

1. The strength of given NaOH solution = ………………..N
2. The amount of NaOH present in 250 ml of the given solution =…………….gms

Viva voce:

1. What will be the shape of the graph if we titrate strong acid with weak base?
2. What is the principle of the conductometric titration?
3. What are the precautions for the experiment?
4. How can the end point be detected?

EXPERIMENT:-12

AIM-To determines the amount of sodium and potassium in given water sample by flame photometer.

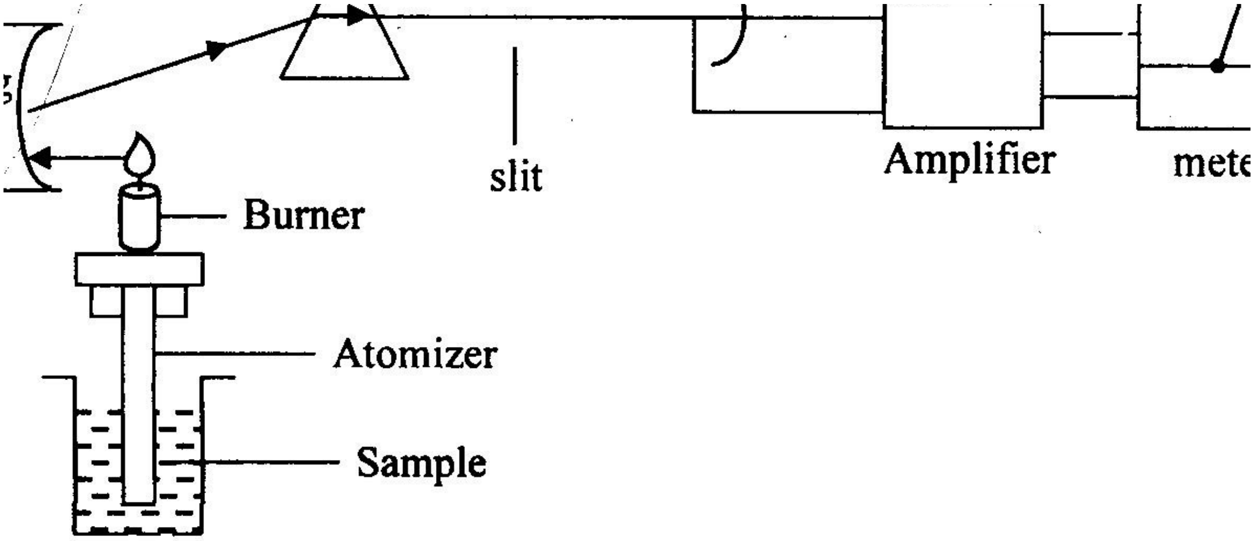
Requirement-NaCl solution, KCl solution, flame photometer, micropipette, beaker, etc.

Theory-Metal present in sample can be analyzed by flame photometer, on the basis of principle“When the solution of metallic salt is sprayed into flame, solvent get vaporized, leaving behind salt”. Salt get vaporized leaving behind constituent atoms. Some of the metal atom gets excited to higher energy state. Exited atoms emit radiations, which are characteristics of the metal atom.

Efficiency of formation of excited atoms into flame is low due to incomplete combustion or formation of molecular species. Due to this reason, the technique cannot be used for analysis of all metals.

Only those metals, which has easily excited flame spectrum have sufficient intensity for detection by photocell.This technique is used for analysis of Na, K, Ca, Li,etc.

Flame photometer are generally used for the analysis of alkali and alkaline earth metals(NA,K,Ca,Li)because these have easily excited flame spectrum of sufficient intensity for detection by photocell.



Flame spectrophotometer (Block diagram)

1. Preparation of standard Sodium solution- Prepare 1000 ppm solution of NACl by dissolving 2.542 gm of NaCl in one litre volumetric flask in distilled water. This is stock solution.
2. Preparation of standard Potassium solution - Prepare 1000 ppm solution of KCl by dissolving 1.909 gm of NaCl in one liter volumetric flask in distilled water. This is stock solution.
3. Further dilute the stock solution by distilled water to obtain the solution of different concentrations.(100 ppm solution is prepared by diluting it 10 times.)Prepare at least five solutions of various concentrations ranging from 100 ppm to 10 ppm.
4. Preparation of calibration curve-To obtain the calibration curve pass air into the atomizer, and the suction thus produced draws a solution of the sample through capillary into atomizer and mixed with oxidant and fuel gas. Burn the mixture in the burner. The radiations resulting from the flame pass through special isolator, which allow only characteristic radiations of element detected by photocell. Plot the calibration curve between optical reading along y-axis and concentration in ppm along x-axis.With the help of calibration curve we can find out the concentration of unknown sample.

The characteristic wave length of emitted radiations-Sodium-589 nm, Potassium-766, Calcium-622, Lithium-671, Barium-553.6

Precautions-

1. For preparation of stock solution, distilled water is to be used.
2. Ratio of fuel gas and oxidants should be proper.
3. For dilution micropipette should be used.

Viva-voce

1. Which type of elements can be analyzed by flame photometer, and why?
2. How flame photometer is related to emission spectroscopy?
3. What is the function of photocell?
4. What are the applications of flame photometry?

EXPERIMENT: - 13

AIM: - To determine the percentage of iron in an iron ore by Redox titration using an n-phenyl Anthranilic acid as internal indicator.[i]

CHEMICALS USED: - Potassium dichromate, 5% stannous chloride, conc. Sulphuric acid HCL, Mercuric chloride an n- phenyl Anthranilic acid as indicator.

APPARATUS REQUIRED: - Beaker, titration flask, conical flask, pipette, funnel, measuring cylinder.

PRINCIPLE: - The estimation of iron ore or alloy can given by potassium dichromate titrations. The common iron ores are Hematite, Magnetite, and Siderite. When these ores or alloys are dissolved the iron is usually obtained in the form of Fe^{3+} Ions present in the ore sol. are converted to Fe^{2+} Ions and then the resulting sol. is titrated against standard potassium dichromate sol. in the presence of acid. The reducing agent used for converting Fe^{3+} to Fe^{2+} is metallic Zinc or stannous chloride.

Theory:- $K_2Cr_2O_7$ oxidises $FeSO_4$ present in the presence of dil.H_2SO_4

$$Cr_2O_7^{-2} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$$

$$Fe^{2+} \rightarrow Fe^{3+} + e^-$$

PROCEDURE: - To prepare the ore solution: Weigh 2 gm of dry ore and transfer it into 100 ml conical flask. Add 15 ml conc. HCl to it. Warm these contents gently until the ore get dissolve. Transfer the contents into 250 ml of round bottom flask and make up the sol. to 250 ml.

Reduction of Fe^{3+} to Fe^{2+} In ore solution:

1. Take 20 ml of above sol. in conical flask.
2. Add 5 ml of conc. HCL and heat the sol. to boiling for 1-3 min.
3. Add $SnCl_2$_Solution to the hot sol. drop wise till the yellow color of the sol. gets discharged.
4. Now cool down the sol. under tap water and dilute 100 ml with water.

Titration with $K_2 Cr_2 O_7$::

1. Rinse and fill the burette with N/10 potassium dichromate.
2. Add few drops of indicator to the sol. in conical flask.
3. Titrate it with pot. Dichromate till the color changes from green to purple
4. Take at least 3 concordant readings.

OBSERVATION TABLE: -

Volume of water sample taken for titration = 100 ml.

S.NO.	Volume of the solution taken in the titration flask	Burette readings		Volume of the titrant used
		Initial Reading	Final Readings	
1 2 3				

RESULT: - The percentage of iron in an iron ore is____%.

GENERAL CALCULATIONS: -

$$(Fe^{2+}) \quad (K_2Cr_2O_7)$$

$$N_1V_1 = N_2V_2$$

$$N1 * 20 = 1/10 * V_2$$

Strength of Fe^{2+} In the sol. = NI * Eq. wt = V2/200 *56 g/L.

So percentage of iron in iron ore = $56V_2/200 * 100/x$.

PRECAUTIONS: -

1. Weighing should be done correctly.
2. Boiling should be avoided.

Viva voce:

1. Why only a very small excess of $SnCl_2$ Is added during the reduction of Fe^{3+}
2. Why the sol. is cooled after reduction is complete.
3. What are the other possible constituents of Iron ore?

GENERAL RULES FOR THE SAFE WORKING OF STUDENTS IN THE LABORATORY

Laboratory Safety

1. **State-approved safety goggles must be worn in the laboratory at all times.** This is required by state health regulation. Failure to observe this requirement will result in your removal from the laboratory. The cost is approximately Rs100 (and trivial in comparison to your eyesight). Safety glasses, etc. are not acceptable. If you already have goggles, you will not be allowed in the laboratory and, consequently, will miss the experiment.
2. **The use of contact lenses in the laboratory is absolutely prohibited.** In the case of a splash or other emergency, they may interfere with the flushing of the eye, you may not be in a position to remove them, and those administering first aid may not know that you are wearing them or might not be able to remove them easily.
3. **You must dress appropriately for the laboratory.** Lab coats are required to be worn. Bare feet, sandals, or other open-toed shoes are not permitted in the laboratory. Shorts and short skirts are likewise not permitted; legs must be covered to below the knees. *Headphones are not allowed in the laboratory.* Failure to observe these requirements will result in your removal from the laboratory. Cotton clothing (including denim) is particularly susceptible to being eaten by acid solution. The laboratory is not a good place to wear your favorite clothes**. Long hair should be tied back.**
4. Learn the location and operation of the safety showers, emergency eyewashes and fire extinguishers in the laboratory. In the case of spill onto a person or clothing, the immediate action should be to flush with water and lots of it. Do not hesitate to yell for help. Report accidents to your instructor. He/she has been certified to administer first aid. If you are not familiar with the operation of the fire extinguishers, ask your instructor to explain them to you.
5. The fire extinguishers should only be used for real emergencies since the chemicals they contain can cause considerable damage. In any emergency that requires the assistance of the fire department, aid car, or police, sends someone to the stockroom for assistance. Should clothing catch on fire, remain as calm as possible. Walk (do not run) to the safety

shower and pull the ring to douse yourself with water. Alternatively, you may drop to the floor and roll to extinguish the flames.

6. Become familiar with all of the exits from the laboratory. A repeating siren and flashing of the FIRE indicator is the building evacuation signal. If this alarm goes off while you are in the lab, turn off any open flames, grab your valuables, and leave the building as quickly as possible.
7. **Never attempt any unauthorized or unassigned experiments**. Follow the experimental procedures explicitly, checking and double-checking the identity of all reagents before you use them. There are potentially hazardous combinations of chemicals present in the laboratory. If you have an idea for further investigation, discuss it with your instructor.
8. **Clean up spills immediately**. The next person to come along has no way of knowing if the clear liquid or white powder on the lab bench is innocuous or hazardous. Neutralize acid spills with sodium bicarbonate (baking soda) before cleaning them up. Spills of sulfuric acid solutions are particularly hazardous since only the water will evaporate, thereby making the solution more concentrated upon standing.
9. **Never return unused reagents to their storage container**. If you take more than you need, dispose of the excess in the appropriate manner. Use the reagents sparingly—they are expensive and time-consuming to prepare. When taking reagents, transfer the amount you need to a clean beaker or other suitable container for taking the material back to your desk. Never insert a pipette or any other object into a liquid reagent container. Finally, check and double-check the identity of all materials before using them.
10. **Do not pick up hot objects**. Be sure your apparatus is cool before picking it up. Do not point the open end of a test tube or other vessel containing a reaction mixture toward yourself or anyone else. If the procedure calls for you to observe the odor of the contents of a vessel, hold it upright in front of you, gently fan some of the vapors toward your nose and sniff cautiously. Most chemical vapors are at least irritating, and many are quite toxic. Please do not taste any chemicals.
11. **Do not eat, drink or smoke in the laboratory**.
12. **Playing of radios, tapes, CDs is not permitted.** This includes small portable devices used with earphones or headsets.

13. Keep coats, backpacks and other non-essential materials away from areas where people are working.
14. Dispose of all broken glassware and other sharp objects in the cardboard glass disposal boxes. Custodial personnel will stop collecting trash after they find broken glass in the trashcans.
15. Wash hands often when working in lab, and always wash thoroughly before leaving.
16. Use the hood for evaporation of anything other than water. The vapors from your procedure alone may not present a problem but those from all the students in the lab could combine to create a hazard.
17. **Do not leave a Bunsen burner or other heated apparatus unattended.** The person working next to you may not know what is involved with your setup and may be working with a flammable material. Turn off open flames if you must leave your area. Make sure the gas taps are completely off whenever the Bunsen burner is not lit.
18. Waste Disposal Dispose of chemical reagents and other materials properly. The proper disposal of chemical wastes is essential to the health and safety of Institute faculty, staff, students and the surrounding community.
19. Chemical wastes must be managed and discarded in the most responsible and environmentally sound method available.