

**WORLD COLLEGE OF TECHNOLOGY AND
MANAGEMENT,
GURGAON (HARYANA)**



**ENGINEERING CHEMISTRY
CH-103-F
LAB MANUAL**

**I, II SEMESTER
B. Tech.**

EXPERIMENT NO:- 1

AIM:- To prepare urea formaldehyde resin.

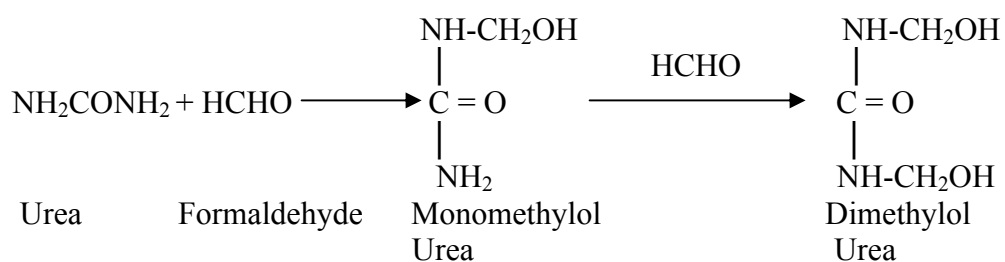
Apparatus required:- Beaker, glass rod, funnel, filter paper and chemical balance.

Chemicals:- Urea, formaldehyde sol., conc. H₂SO₄, distilled water.

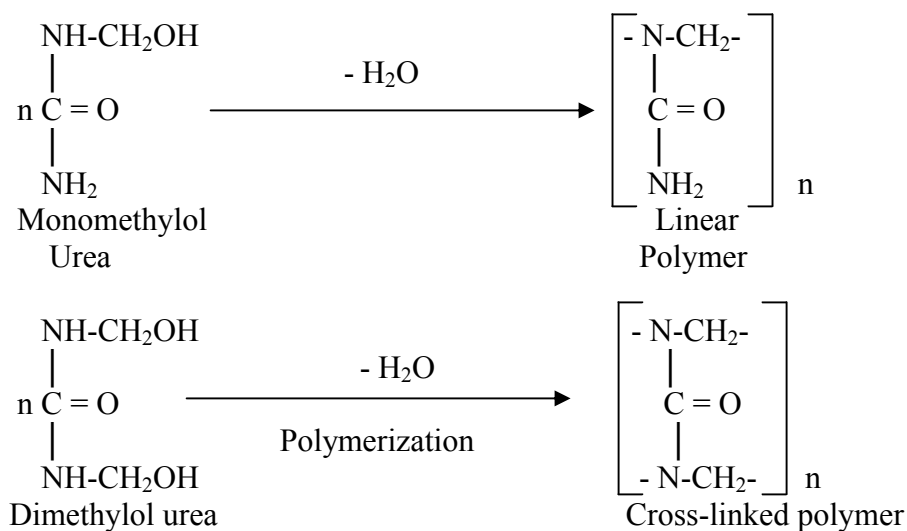
Theory:- Amino resins are condensation products obtained by the reaction of formaldehyde with nitrogen bearing compounds such as aniline, amides for ex:- melamine formaldehyde, urea formaldehyde etc.

Urea formaldehyde is prepared by condensation reaction between urea and formaldehyde in acidic or alkaline medium.

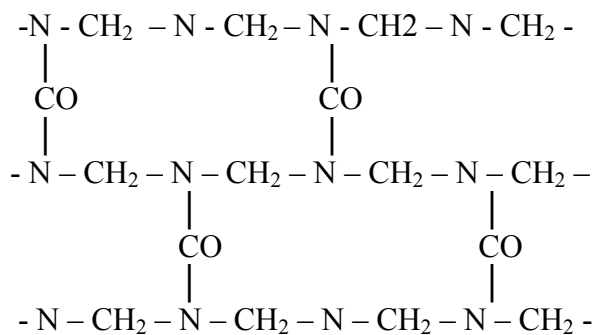
The first product formed during the formation of resin is monomethylol and dimethylol ureas.



Polymerization can take place from mono or dimethylol urea or possibly through both, with the formation of long chains.



A fully cross-linked urea formaldehyde resin can be represented as:-



Urea formaldehyde resin (cross-linked polymer)

Procedure:-

1. Place about 5 ml 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. H₂SO₄, 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. Weight the yield of product

Precautions:-

1. While adding concentrated H₂SO₄, it is better to stay little away from the beaker since the reaction sometimes becomes vigorous.
2. The reaction mixture should be stirred continuously.

Observations:-

Mass of the beaker (W₁) = -----g.

Mass of the beaker with urea formaldehyde (W₂) = -----g.

Therefore mass of urea formaldehyde (W₂ – W₁) = -----g.

Result:- The yield of urea formaldehyde = -----g

Properties:

1. They have good electrical insulating properties.
2. They are resistant to oil, grease and weak acids.
3. They are hard, resist abrasion and scratching.
4. They have good adhesive properties.

Uses:-

1. They are used adhesive applications for the production of plywood and laminating .
2. They are used for the manufacture of cation exchange resins.
3. These also find use in the manufacture of electrical switches, plugs and insulating foams.

4. Their applications also include the treatment of textile fibers for improving their shrink and crease resistance.

EXPERIMENT NO:- 2

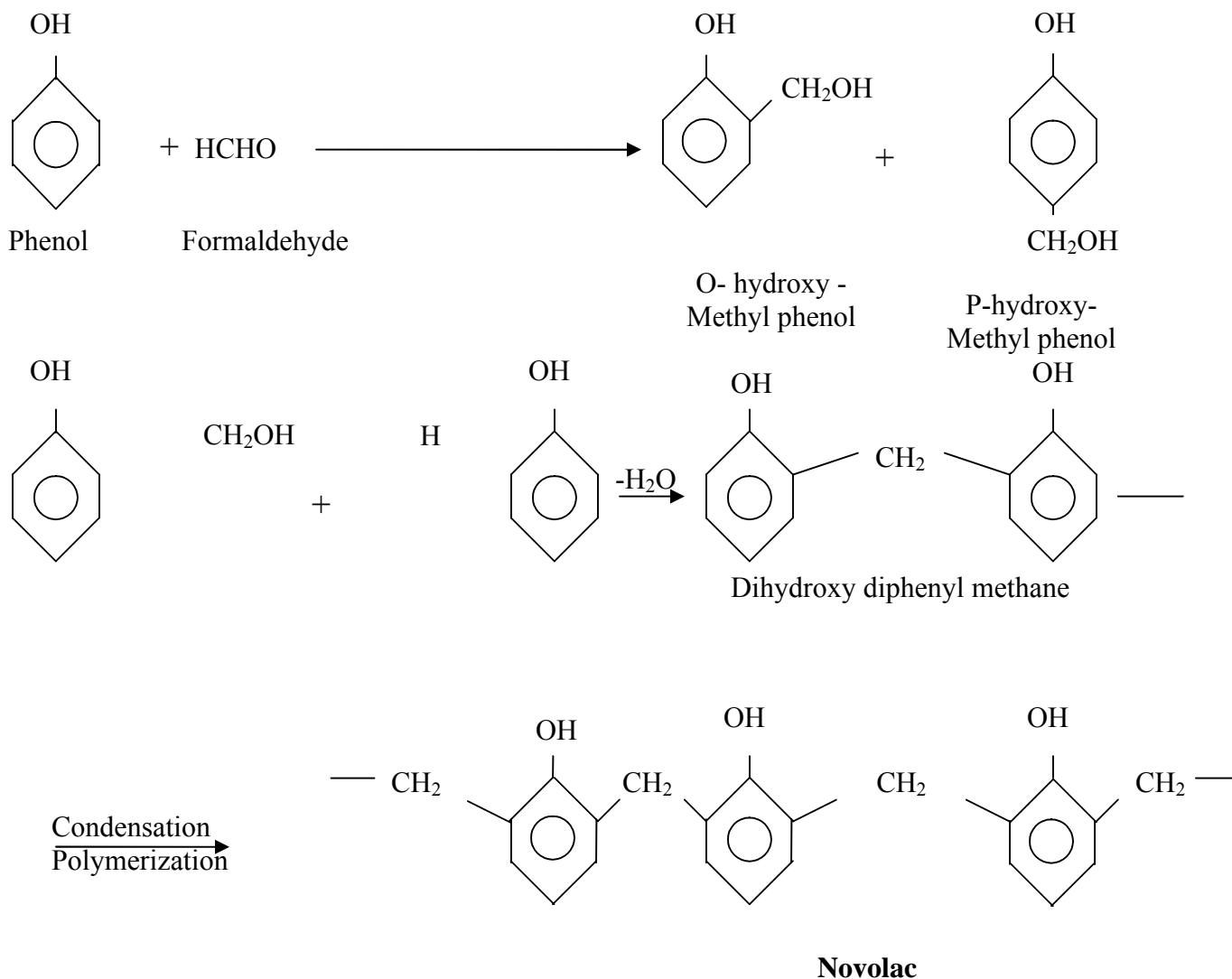
AIM:- To prepare phenol formaldehyde resin.

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

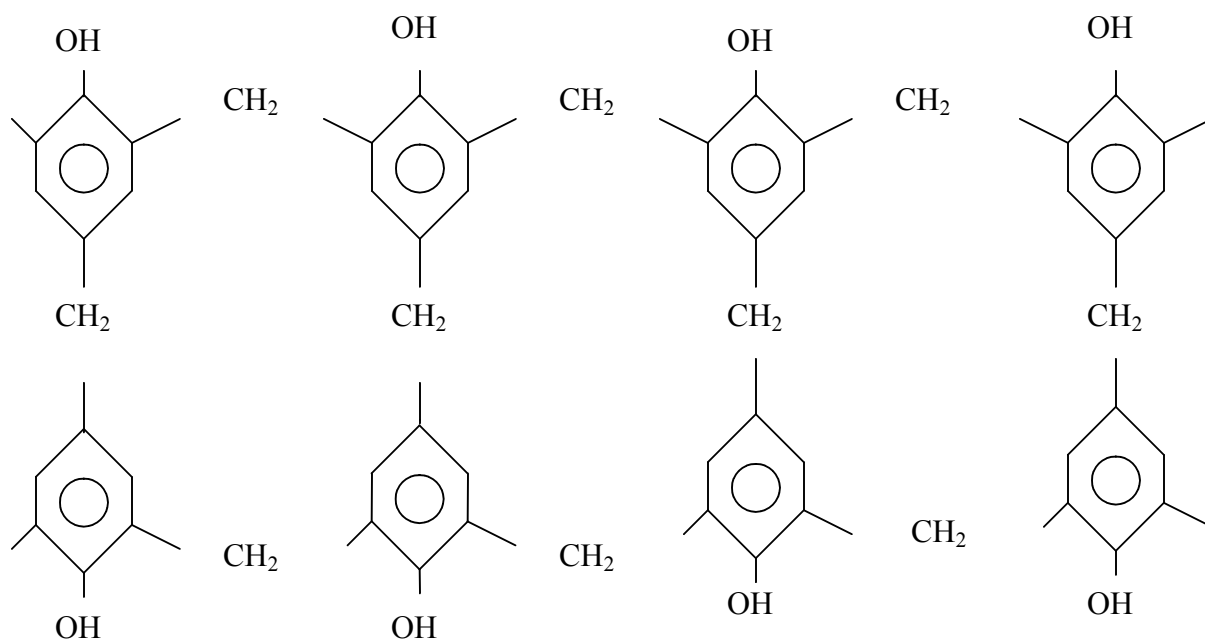
Chemicals:- Phenol formaldehyde, conc. HCl, glacial acetic acid, distilled water.

Theory:- Phenolic resins are condensation polymerization of phenolic derivatives (like phenol, resorcinol) with aldehyde (like formaldehyde, furfural). Most important member of this class is bakelite or phenol formaldehyde resin.

Phenol formaldehyde is prepared by condensing phenol with formaldehyde in presence of acidic or alkaline catalyst. The initial reaction result in the formation of o- and p-hydroxy methyl phenol, which



react to form linear polymer novolac. During moulding hexamethylene tetramine [(CH₂)₆N₄] is added which convert the fusible novolac in to hard infusible and insoluble solid of cross – linked structure known as Bakelite.



BAKELITE

Procedure:-

1. Place 5 ml of glacial acetic acid and 2.5 ml of 40% formaldehyde solution in a 100 ml beaker .
2. Add 2 g of phenol to it.
3. Wrap a cloth loosely round the beaker. Add a few ml of conc. HCl in to the mixture carefully and heat it slightly.
4. A large mass of plastic pink in colour is formed.
5. A residue is washed with water and filtered.
6. The product dried and yield is weighed.

Precautions:-

1. While adding conc. HCl, it is better to stay little away from the beaker since the reaction sometimes becomes vigorous.
2. The reaction mixture should be stirred continuously.

Observations:-

Mass of the beaker (W1) = -----g.

Mass of the beaker with phenol formaldehyde (W2) = -----g.

Therefore mass of phenol formaldehyde (W2 – W1) = -----g.

Result: - The yield of phenol formaldehyde is = -----g

Properties:-

1. Phenol formaldehyde moulding resins have excellent heat resistance.
2. These have high dimensional stability.
3. Phenolic resins have good dielectric properties.
4. They have hard, rigid and scratch resistant.

- Uses:-**
1. They are used for making electric insulator parts like switches, plugs, switch board, heater, handles etc.
 2. These are also used in varnishes, paints and protective coatings.
 3. These are used in the protection of ion exchange resins for water softening.
 4. Phenolic resins are used for improving impregnating paper, wood and other fillers.

VIVA – VOCE FOR EXPERIMENT NO: - 1 & 2

Q1.what is the difference between thermosetting and thermo plastic space ?

Ans:in thermo plastic, polymer there is no linkage between chains as a result these can easily moulded by heating in thermosetting plastics , there is extensive cross linkage between different polymer chains forming 3D network of bonds.

Q2.UF &PF raisins are thermo plastics or thermo settings?

Ans: Thermosetting as they cannot be moulded in any other shape once made by heating.

Q 3.Which one owt of UF & PF raisins is formed faster? Why it is so ?

Ans: PF raisin is formed faster than UF raisin because phenol raisin is prepared in two stepwhere as UF raisin is formed in three/four steps and it includes formation of many products.

Q 4. Give few applications each of UF & PF.

Ans: UF is used for electrical fittings, adhesives, plywoods& lamination products.PF is used for domestic plugs and switches, cooker handelsect.

Q 5.What are poly meric composites, give exampels.

Ans: UF resins deu to their color and lightness are used to make light colored objects and deu to their hardness they are used for making grindings .

Q6.Do you think the resins you are preparing in the lab will have the above application on these must be suitabeli compounded by adding activities.

Ans: The resins we have prepared will not have the above applications because they are in raw form and they are converted into different forms of suitable method .

EXPERIMENT NO: - 3

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

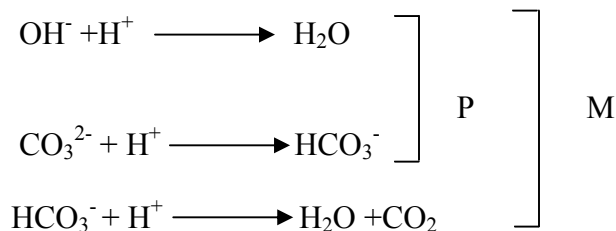
Apparatus required:- Burette, pipette, conical flask, beaker, measuring flask and droppers.

Chemicals:- N / 50 HCl solution, phenolphthalein and methyl orange indicator.

Theory:- Alkalinity is a measure of capacity of water to neutralize acids. Alkalinity in water is due to the presence of following ions:-

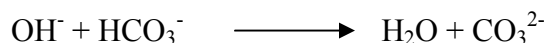
- OH^-
- CO_3^{2-}
- HCO_3^-

When standard acid solution is added to alkaline water following reactions takes place:-



So, in order to find out alkalinity in water, alkaline water is titrated with standard acid solution using phenolphthalein and methyl orange indicator. Phenolphthalein is sensitive to OH^- and CO_3^{2-} where as methyl orange is sensitive to all three ions as indicated in the reactions.

Further, hydroxide and bicarbonate ions can not exist together in water due to the following reaction:-



So there exist five possibilities of alkalinity in water on the basis of conc. of various ions in water as tabulated in the table.

TABLE SHOWING DIFFERENT CASES OF ALKALINITY IN WATER

Titration result	Volume of acid used For neutralization of OH^-	Volume of acid used For neutralization of CO_3^{2-}	Volume of acid used For neutralization of HCO_3^-
$P = 0$	Absent	Absent	M
$P = M$	$P = M$	Absent	
$P = \frac{1}{2} M$	Absent	$2P$ or M	Absent
$P > \frac{1}{2} M$	$2P - M$	$2(M - P)$	Absent
$P < \frac{1}{2} M$	Absent	$2P$	$M - 2P$

Here P = Phenolphthalein end point
M = Methyl orange end point

Procedure:-

1. Pipette out 25 ml of given water sample in a conical flask and few drops of phenolphthalein indicator to obtain pink colour.
2. Titrate the resulting solution with std. N / 50 HCl solution till pink colour disappears.
3. Now add few drops of the methyl orange indicator in the same conical flask to obtain yellow colour.
4. Titrate further with std. HCl solution till pink colour appears.
5. Repeat these steps to obtain three concordant values.

Precautions:-

1. The burette, pipette and conical flask should be rinsed with distilled water.
2. After rinsing with distilled water burette and pipette should be rinsed with desired solution.
3. Funnel should be removed from the burette during titration.

Observation table:-**Titration of water sample with standard N / 50 HCl solution**

Sr. No.	Volume of water sample taken (ml)	Burette readings				
		Initial reading (x) (ml)	Reading up to P end point (y) (ml)	Volume of acid used up to P end point (y-x)= p (ml)	Reading up to M end point (Z) (ml)	Volume of acid used up to M end point (Z-x)=M (ml)
1.						
2.						
3.						
Concordant reading						

Here P = Phenolphthalein end point = $y - x = \text{-----ml}$

M = Methyl orange end point = $Z - x = \text{-----ml}$

Calculations:-

After locating the types of alkalinity use following equations for calculating alkalinity for each ion.

$$N_1V_1 = N_2V_2$$

Where N_1 = Normality of OH^- ions / CO_3^{2-} ions / HCO_3^- ions

$V_1 = \text{Volume of water sample} = 25 \text{ ml}$
 $N_2 = \text{Normality of std. HCl solution} = N / 50$
 $V_2 = \text{Volume of HCl used for each ion} = \text{-----ml}$

Then
$$N_1 = \frac{N_2 V_2}{V_1}$$

Alkalinity = Normality \times equivalent weight of CaCO_3 (g/l.)

$$= N_1 \times 50 \text{ g/l.}$$

Alkalinity = Normality $\times 50 \times 1000$ (mg/l. or ppm)

$$= N_1 \times 1000 \times 50 \text{ ppm}$$

Alkalinity due to OH^- in terms of $\text{CaCO}_3 = \text{-----ppm}$

Alkalinity due to CO_3^{2-} in terms of $\text{CaCO}_3 = \text{-----ppm}$

Alkalinity due to HCO_3^- in terms of $\text{CaCO}_3 = \text{-----ppm}$

Total alkalinity in terms of $\text{CaCO}_3 = \text{-----ppm}$

Result:-

Alkalinity due to OH^- in terms of $\text{CaCO}_3 = \text{-----ppm}$

Alkalinity due to CO_3^{2-} in terms of $\text{CaCO}_3 = \text{-----ppm}$

Alkalinity due to HCO_3^- in terms of $\text{CaCO}_3 = \text{-----ppm}$

Total alkalinity in terms of $\text{CaCO}_3 = \text{-----ppm}$

VIVA VOCE

Q 1; - What do u mean by alkanity of water?

Ans. It is defiend as the amount of standard acid in mg required to neutralizeone litre of given water sample.

Q 2 :Which constituent are responsible for alkalinity in water?

Ans;- It is due to presence of hydroxide ion, carbonate ions and bicarbonate.

Q 3 ;- Why two indicators are used in determining alkalinity of water?

Ans;- It is because of different Ph of water due to different ions causing alkalinity. The two indicators give end point at different PH i.e. phenolphthalein at PH at 8-3 and methyl orange 3 to 4.5.

Q 4; - How much alkalinity is permissible for drinking water?

Ans;- It should be less than 100 ppm.

Q 5 ;- On which factors the use of acid indicator in a titration depends ?

Ans;- It depends upon the PH of the solution.

EXPERIMENT NO:- 4

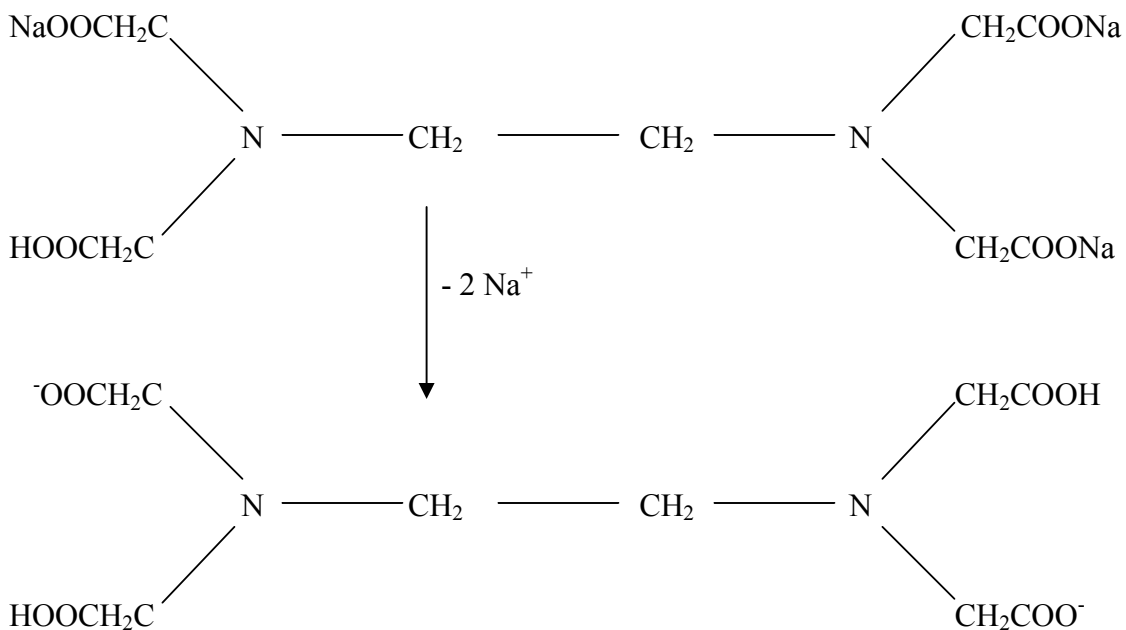
AIM:- To determine Ca^{2+} and Mg^{2+} and total hardness of water using EDTA solution.

Apparatus required:- Burette, pipette, conical flask, beaker, measuring flask and droppers.

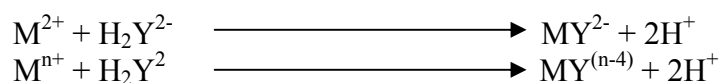
Reagent required:-

1. Standard hard water (N/50, 1 mg CaCO_3 / 1ml water i.e, 1000 ppm.)
2. EDTA solution (N/50)
3. Ammonium chloride, ammonium hydroxide buffer (pH – 10)
4. Eriochrome Black – T indicator solution.
5. Di-ethyl amine.
6. Calcon indicator solution.

Theory:- Disodium salt ethylenediamine tetra acetic acid (EDTA) ionizes in water to give 2Na^+ ions and a strong chelating ligand.

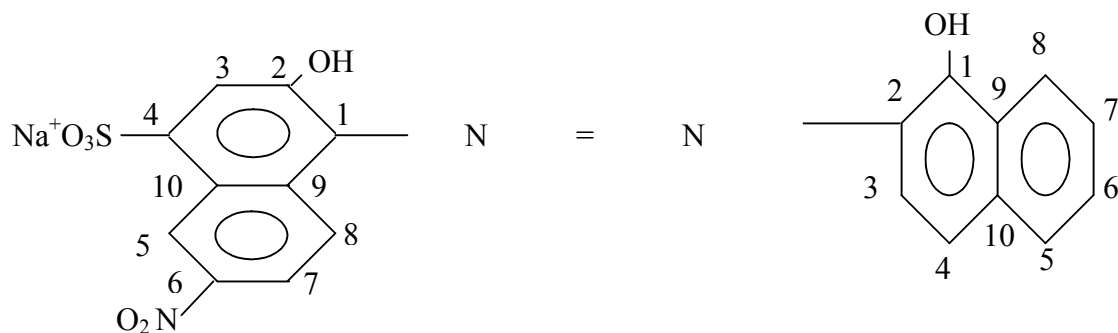


For simplicity it can be represented by H_2Y^{2-} ions. It forms complexes with Ca^{2+} ions and Mg^{2+} ions and other divalent or higher valent cations as represented by the equation given below:-



Complexes formed with divalent metal ions are stable in the pH range 8 – 10 .

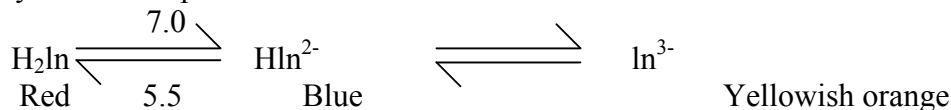
In the determination of hardness of water eriochrome black – T indicator is used.



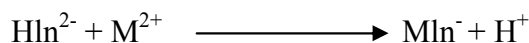
ERICHROME BLACK – T

[Sodium – 1 – (1 – hydroxy – 2 – naphthylazo) – 6 – nitro – 2 – naphthol – 4 – sulphonate]

For simplicity it can be represented as $\text{Na}^+ \text{H}_2\text{In}^-$. This indicator show different colour in different pH range.

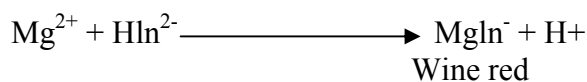


In the pH range 7 – 11, the indicator forms weak complexes with metal ions their complexes give wine red colour to the solution.

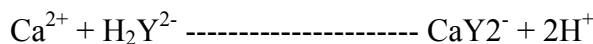


So during hardness determination pH of the solution is maintained at 10 using buffer solution.

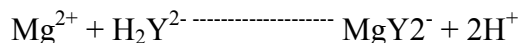
When a small amount of indicator is added to the hard water sample where pH has been controlled by the addition of buffer solution, the indicator react with Mg^{2+} ions present in water to produce Mg indicator complex and impart wine red colour to the solution.



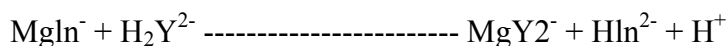
Now during titration EDTA (H_2Y^{2-}) is added, it forms complexes with free Ca^{2+} as Ca – EDTA complex is more stable.



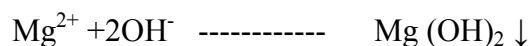
Remaining free Mg^{2+} ions are then form complex with EDTA Mg – EDTA less stable then Ca – EDTA complex.



Mg – EDTA complex is more stable then Mg – indicator complex so on further addition EDTA Mg – indicator complex dissociates and formation of more stable Mg – EDTA complex take place this result in the liberation of free indicator in the solution which in this pH range give blue colour.



Thus we obtain the end point of the titration. This titration gives the total hardness of water sample. If diethyl amine is added to water sample it raises the pH to about 12.5 at this pH Mg^{2+} precipitates as $\text{Mg}(\text{OH})_2$



So Ca^{2+} hardness can be determined using calcon as indicator in the resulting solution and titrating with std. EDTA solution colour changes from pink to blue at the end point.

Titration of resulting solution gives calcium hardness. Mg hardness can be obtained by subtracting calcium hardness from total hardness.

Procedure:-

(a) Standardization of EDTA solution with std. hard water.

1. Pipette out 25 ml of std. hard water into a conical flask and add 40 ml distilled water and 2 ml of buffer solution to it.
2. To the resulting solution add 2 drops of indicator Erichrome black – T it should give wine red colour.
3. Rinse the burette with EDTA and fill it with EDTA solution.
4. Titrate the solution of conical flask with EDTA solution till blue colour appears.
5. Take three concordant readings. (volume of EDTA used = ----- ml).

(b) Determination of total hardness

1. Pipette out 25 ml of std. hard water into a conical flask and add 2 ml buffer and few drops of indicator to it.
2. Titrate the solution of conical flask with std. EDTA solution till blue colour appears.
3. Record volume of EDTA used ----- ml.

(c) Determination of calcium hardness

1. Pipette out 25 ml of std. hard water into a conical flask.
2. Add 3 ml of diethyl amine and add 4 drops of calcon indicator.
3. Titrate the solution of conical flask with std. EDTA solution until colour changes from pink to pure blue.
4. Volume of EDTA used ----- ml.

Precaution :-

1. The burette, pipette and conical flask should be rinsed with distilled water.
2. Redistilled water should be employed for preparing the EDTA solution.
3. The colour change near the end point is very slow and thus should be observed carefully.

Observations :

Table 1

(a) Standardization of EDTA

Titration between standard hard water & EDTA solution

S. No.	Volume of Std. Hard Water (ml)	Initial Reading (ml)	Final Reading (ml)	Volume of EDTA used (ml)
1.	25.0			
2.	25.0			
3.	25.0			

Concordant Reading ----- ml

Table 2

(b) Determination of Total Hardness

Titration between standard EDTA and hard water sample for total hardness

S. No.	Volume of Hard Water (ml)	Initial Reading (ml)	Final Reading (ml)	Volume of EDTA used (ml)
1.	25.0			
2.	25.0			
3.	25.0			

Concordant Reading ----- ml

Table 3

(c) Determination of Calcium Hardness

Titration between standard EDTA and hard water sample for Ca hardness

S. No.	Volume of Hard Water (ml)	Initial Reading (ml)	Final Reading (ml)	Volume of EDTA used (ml)
1.	25.0			
2.	25.0			
3.	25.0			

Concordant Reading ----- ml

Calculations :-

a. Standardization of EDTA

$$N_1 V_1 = N_2 V_2$$

N_1 = Normality of EDTA ?

V_1 = Volume of EDTA used

N_2 = Normality of hard water = $N/50$

V_2 = Volume of hard water = 25.0 ml

$$N_1 = N_2 V_2 / V_1$$

$$N_1 = 25/50 \times V_1 = 1/2 V_1 \text{ eq./lt}$$

(a) Total hardness

$$N_1 V_1 = N_3 V_3$$

N_1 = Normality of EDTA ?

V_1 = Volume of EDTA used

N_3 = Normality of hard water ?

V_3 = Volume of hard water = 25.0 ml

$$N_3 = N_1 V_1 / V_3 = N_1 V_1 / 25 \text{ eq./lt}$$

Total hardness in terms of CaCO_3 = $50 \times N_3 = N_1 V_1 / 25 \times 50 \text{ g/lt}$

Total hardness in terms of CaCO_3 in ppm = $50 \times N_3 \times 1000 \text{ ppm}$.

(b) Calcium and Magnesium hardness

$$N_1 V_1 = N_4 V_4$$

N_1 = Normality of EDTA

V_1 = Volume of EDTA used

N_4 = Normality of Ca hard water

V_4 = Volume of hard water = 25.0 ml

$$N_4 = N_1 V_1 / V_4 = N_1 V_1 / 25 \text{ eq./lt}$$

Ca hardness in terms of CaCO_3 = $50 \times N_4$

Ca hardness in terms of CaCO_3 in ppm = $50 \times N_4 \times 1000 \text{ ppm}$

Mg hardness in terms of CaCO_3 = Total hardness – Ca hardness

$$= \text{-----} \text{ ppm}$$

Result:-

Calcium hardness of water sample in terms CaCO_3 = -----ppm

Magnesium hardness of water sample in terms CaCO_3 = -----ppm

Total hardness of water sample in terms CaCO_3 = -----ppm

Viva voce

Q1. What is hardness of water

Ans. Hardness is defined as the characteristics which prevents the lathering of soap.

Q2. How hardness is expressed?

Ans. Hardness is expressed in terms of CaCO₃ equivalent.

Q3. Why hardness is expressed in terms of CaCO₃ equivalent?

Ans. It is due to two reasons: (i) Mol. wt. of CaCO₃ being 100 make calculation easier. (ii) It is the most insoluble compound precipitated out in water treatment.

Q4. How water becomes hard?

Ans. Water becomes hard due to its action on rock and minerals. This may involve hydrolysis, dissolution, disintegration, oxidation etc. of minerals by water.

Q5. What are various units of hardness?

Ans. Hardness of water can be expressed in following units:

- (I) Parts per million (ppm)
- (II) Milligram per litre (mg/l)
- (III) Degree Clarke (*C)
- (IV) Degree French (*F)

Q6. Name some methods for removing permanent hardness of water.

Ans. There is (i) lime-soda (L-S) method (ii) zeolite method (iii) ion-exchange resin method

Q7. Which of the above methods is best for removing permanent hardness?

Ans. Ion-exchange resin method is best as it gives water for almost zero hardness.

Q8. Give few disadvantages of using hard water.

Ans. Some of the disadvantages of using hard water is:

- (i) It takes more time and consumes more soap for washing clothes.
- (ii) Use of hard water results into scale and sludge formation inside boilers
- (iii) It takes more time for cooking with hard water.

Q9. Why does hard water not lather with soap?

Ans. Ca⁺² and Mg⁺² ions of hard water form insoluble soaps on treating hard water with soap
 $2\text{RCOONa (soap)} + \text{M}^{+2} \text{ (from hard water)} \rightarrow (\text{RCOO})_2 \text{M} + 2\text{Na}^+$ (metallic soap ↓ white ppt or scum)
Lather is only formed when all hardness causing metal ions are removed from water.
So more soap is consumed.

Q10. What is a chelating agent?

Ans. It is a poly dent ate legant which coordinates with metal ions to form a stable ring complex
The complex so formed is called a chelate .eg.
EDTA is a chelating hexadentate legant.

EXPERIMENT NO. – 5

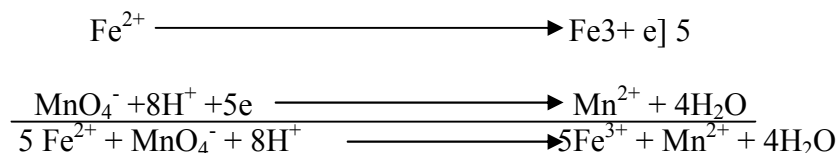
AIM: - Determination of the amount of iron in an iron ore solution by KMnO_4 .

Apparatus required: - Burette, pipette, conical flask, beaker, measuring flask and droppers.

Reagents Required:-

1. Standard potassium permanganate solution (N/20).
2. Copper sulphate solution.
3. Ammonium thiocyanate solution.
4. Dilute sulphuric acid.
5. Granulated zinc or zinc dust.

Theory:- A known volume of the iron ore solution prepared in H_2SO_4 is reduced with Zn and H_2SO_4 . After removal of excess of zinc, the solution is diluted to a definite volume. A suitable aliquot of this solution is then titrated with standard KMnO_4 solution.



KMnO_4 acts as self – indicator and appearance of light pink colour shows the end point.

Procedure:-

1. Weigh out Fe ore sample and dissolved in sufficient dilute H_2SO_4 making volume 250 ml.
2. Take 50 ml of above Fe ore solution in conical flask. Add 5g granulated Zn and 2 or 3 drops of CuSO_4 .
3. Place a short funnel in the mouth of conical flask and add about 25 ml of dilute H_2SO_4 (6–8N). Slightly warm and allow the reaction to continue with shaking until the solution appear pale green.
4. Now test for absence of Fe^{+++} ion by taking a drop of above solution in a test tube containing NH_4SCN solution till it does not give blood red colour.
5. Filter the solution through a plug of glass wool placed in the neck of a funnel in to a 100ml measuring flask.
6. Rinse the conical flask to 2-3 times with 5ml portion of dilute H_2SO_4 and pass the same through filter. Make the volume up to the mark of measuring flask with distilled water.
7. Pipette out 25 ml of this solution in to a titration flask and add about 5ml of dilute H_2SO_4 and titrate it against std. KMnO_4 solution until a light pink colour appears in the reaction mixture.
8. Take three concordant readings.

Precautions:-

1. All the zinc used for reduction should be removed before titration. If zinc dust has been used for reduction, it is better to boil the solution to completely dissolved the zinc.
2. After the reduction is complete, the determination should be made as quickly as possible to avoid atmospheric oxidation of Fe^{2+} to Fe^{3+}

Observation:-

Mass of iron ore = W g
Volume of iron ore solution prepared = 250ml
Volume of ore solution taken = 50ml
Volume made after reduction = 100ml
Volume of reduced solution taken for each titration = 25.0ml
Strength of $\text{KMnO}_4 = \text{N}/20$

Table

Titration of Fe ore solution with N/20 Std. KMnO_4 solution

S. No.	Volume of Fe^{+++} taken (ml)	Burette Reading		Volume of KMnO_4 used (ml)
		Initial Reading (ml)	Final Reading (ml)	
1.	25.0			
2.	25.0			
3.	25.0			

Calculation:-

$$N_1 V_1 = N_2 V_2$$

$N_1 =$ Normality of Fe^{2+}

$V =$ Volume of iron ore after dilution = 25.0ml

$V_1 =$ Volume of iron ore solution before dilution = $25/2 = 12.5\text{ml}$

$N_2 =$ Normality of KMnO_4 solution = $\text{N}/20$

$V_2 =$ Volume of KMnO_4 used

$$N_1 = N_2 V_2 / V_1$$

$$N_1 = 1/20 \times V_2 / 12.5 \text{ eq./lt}$$

Mass of iron in one liter = $N_1 \times 56\text{g/lt}$

Mass of iron in 250ml ore solution = $N_1 \times 56/4\text{g}$

Percentage of iron in iron ore = $\frac{\text{Mass of iron} \times 100}{\text{Mass of iron ore}} = N_1 \times 14 \times 100/W$

Result:- Percentage of iron in given iron ore = -----

VIVA VOCE

Q1 Define an ore ?

Ans .ore is combined form of a metal existing in earth 's crust from which metal can be profitably extracted by metallurgical operation .

Q2. Give few important ore's of iron:-

Ans .some important ore's of iron are

- 1) magnetite or load stone (Fe_3O_4)
- 2) haematite or red iron ore (Fe_2O_3).
- 3) Iron pyrite(FeS_2)
- 4) Siderite (FeCO_3)

Q3 what is the difference between an ore and mineral ?

Ans .mineral is the combined form of metal in which it occurs in earth's crust or rocks. A mineral which metal can be obtained

Commercially is called an ore .So ,all ores are minerals but all minerals are not ores .

Q4 How is iron ore solution prepared ?

Ans. Iron ore is prepared by dissolving the ore in an acid like
HCL or H_2SO_4 .

Q5 . Define oxidation and reduction .

Ans .Process involving loss electron is called oxidation while that in which electrons are gained is termed reduction .

Q6. what are redox titrations?

Ans .A reaction in which oxidation and reduction take place

Simultaneously is called redox reaction .Titrations involving redox reactions are called redox titrations.

Q7. Define an oxidizing agent .

Ans. A substance which oxidises other substance by getting reduced itself is called an oxidising agent .

Q8. What is reducing agent ?

Ans. A substance which reduces other substance a itself gets oxidized in doing so is termed as reducing agent .

Q9. Mention some other reagents which can be used for reduction of iron ore solution.

Ans. 1) Hydrogen sulphide

- 2) sulphurous acid and sulphur dioxide .

EXPERIMENT NO:-6

AIM:- Determination of dissolved oxygen in the given sample of water.

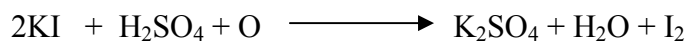
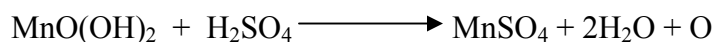
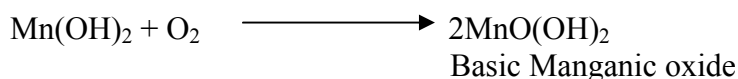
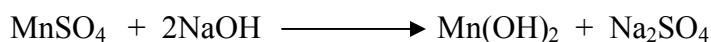
Apparatus:- Burette, pipette, conical flask, beaker, measuring flask and droppers.

Chemicals:- $\text{Na}_2\text{S}_2\text{O}_3$ (N/100), MnSO_4 solution, KI, Starch, conc. H_2SO_4 .

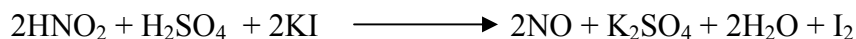
Theory:- Oxygen itself is not a pollutant in water but its deficiency is an indicator of several types of pollution in water.

Dissolved oxygen (DO) is determined by Winkler's method or iodometric titration. The dissolved oxygen in water oxidizes KI and an equivalent amount of iodine is liberated. This iodine is titrated against a standard hypo solution. However since dissolved oxygen in water is in molecular state and is not capable of reacting with KI therefore an oxygen carrier such as manganese hydroxide is used.

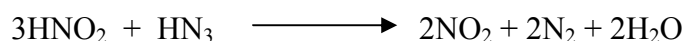
The method involves introducing a conc. solution of MnSO_4 , NaOH and potassium iodide, azide reagent, in to the water sample. The white precipitate of $\text{Mn}(\text{OH})_2$ formed, is oxidized by oxygen in water sample to give a brown precipitate of basic manganic oxide $\text{MnO}(\text{OH})_2$. This $\text{MnO}(\text{OH})_2$ in acidic medium dissolves and liberates free iodine from the added KI in an equivalent amount of dissolved oxygen in water sample. This liberated I_2 is then titrated against $\text{Na}_2\text{S}_2\text{O}_3$ using starch as an indicator. The reactions involved are:-



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



Thus to destroy nitrite, sodium azide is used.



Procedure:-

1. A known amount of sample water(say 250ml) is taken in a stoppered bottle avoiding contact with air.
2. Add 0.2ml of MnSO_4 solution it by means of a pipette, dipping the end well below the surface of water. Also add 2ml of alkaline iodide – azide solution to it.
3. Stopper the bottle and shake thoroughly. Allow the brown precipitate of $\text{MnO}(\text{OH})_2$ formed, to settle down.
4. When some portion of the liquid below the stopper is clear, add 2ml of conc. H_2SO_4 with the help of pipette. Stopper and mix till the precipitate is completely dissolved. The characteristics brown colour of iodine is produced.
5. Transfer 100ml of above solution in a 250ml flask with a pipette. Titrate the liberated I_2 with standardized sodium thiosulphate solution until the sample solution becomes pale yellow.
6. Add 2ml of starch solution the solution will turn blue.
7. Continue titration till the blue colour disappears.
8. Repeat to get another reading.

Precautions:-

1. The water should be taken in a stoppered bottle very carefully without trapping air bubbles, which could raise oxygen level by aerating the sample.
2. MnSO_4 and alkaline iodine – azide solutions are added to the water sample just below the surface of water.
3. Whole of the precipitate of $\text{MnO}(\text{OH})_2$ should be dissolved in H_2SO_4 .

Observations:-

Normality of $\text{Na}_2\text{S}_2\text{O}_3 = N_2 = N/100$

Volume of the water sample taken for titration = $V_1 = 25.0\text{ml}$

Table
Titration of water sample containing dissolved O_2 with $N/100 \text{Na}_2\text{S}_2\text{O}_3$ or hyposolution

S. No.	Volume of Solution taken in titration flask (ml)	Burette Reading		Volume of $\text{Na}_2\text{S}_2\text{O}_3$ used (ml)
		Initial Reading (ml)	Final Reading (ml)	
1.	25.0			
2.	25.0			
3.	25.0			

Calculations:-

$$N_1V_1 = N_2V_2$$

N_1 = Normality of DO in water sample = ?

V_1 = Volume of water sample containing dissolved oxygen = 25.0ml

N_2 = Normality of $\text{Na}_2\text{S}_2\text{O}_3$ = $N/100$

V_2 = Volume of $\text{Na}_2\text{S}_2\text{O}_3$ = -----ml

$$N_1 = N_2V_2/V_1 = 1/100 \times V_2/25$$

Dissolved Oxygen in water sample in g/l = $N_1 \times 8$ g/l

Dissolved Oxygen in water sample in ppm = $N_1 \times 8 \times 10^3$ ppm

Result:- The amount of dissolved oxygen in water = -----ppm

VIVA VOCE

Ques: - Give the significance of dissolved oxygen.

Ans:-1. It is essential for survival of aquatic life

2 The D.O. content predicts purity and the ability of stream to purify itself through bio chemical process.

3 Higher amount of D.O. in water is harmful for boilers since it causes corrosion of boiler.

Ques:-what is the role played by manganous sulphate in D.O. determination?

Ans Dissolved oxygen in molecular state can not oxidise KI to liberate I_2 so it is converted into atomic oxygen with the use of manganous sulphate through the process of D.O. fixation.

Ques:-Which indicator is used in iodometric titrations?

Ans: - Freshly prepared 1% starch solution is used as indicator.

Ques:- Why air bubbles are removed from water sample for D.O. determination ?

Ans: - The oxygen of air present inside the bubbles will also participate in reaction there by giving higher results for D.O.

Ques:-What is the change in oxidation state of Mn during D.O. fixation?

Ans:-Oxidation state of Mn changes from +2 to +4.

EXPERIMENT NO:- 7

AIM:- Determine the viscosity of diesel oil by Red Wood viscometer No.1

Requirement:- Red Wood viscometer No.1, stop watch, diesel oil.

Theory:- The viscosity of diesel oil can be determined by measuring the rate of flow oil through a capillary tube kept at a uniform temperature. But in case of diesel oil specific viscosity is generally determined by measuring the time taken for a given quantity of oil to flow through an orifice or jet of standard dimensions. Under standard conditions, these are types of viscometer redwood, saybolt and viscometer are in common use.

The redwood viscometer measure viscosity in empirical units and not in absolute unit. The formula use to calculate viscosity of diesel oil in given below:-

$$\frac{\eta_{oil}}{\eta_{water}} = \frac{t_{oil}}{t_{oil}} \times \frac{d_{oil}}{d_{water}}$$

Where:-

η = Viscosity

t = Time

d = density

Procedure:-

1. Clean the viscometer cup and properly dry it to remove any trace of the solvent.
2. Level the viscometer with help of leveling screw.
3. Fill the outer bath with water to determining the viscosity at 80°C and below, for higher temperature, the bath is filled with oil having suitable viscosity at the test temperature.
4. Place the ball valve on the jet to close it and pour the test oil in to the cup up to tip of indicator.
5. Place a clean dry kohlrusch flask or beaker immediately below and directly in line with discharging jet.
6. Insert a thermometer and stirrer in the cup and cover it with a lid.
7. Connect the heater switch to the viscometer. Heat the water filled in the bath slowly with constant stirring. When the oil in cup attains a desired temperature stop the heating.
8. Lift the ball valve and start the stop watch. Oil from the jet flows in to the flask.
9. Stop the stop watch when a lower meniscus of oil reaches the 100ml mark on the neck of the receiving flask (replace the ball valve in position immediately so as to prevent overflow).
10. Record the time taken for 100ml of oil to collect in flask.
11. Repeat the experiment to get the reproducible results and report the mean value in second.

Precautions:-

1. Redwood viscometer should be clean.
2. Stopwatch should be started at accurate time i.e. at which the oil start flow.

3. Stopwatch should be stopped at the time when oil finished.

Observation table:-

Time for dropping light oil (100ml) by Redwood viscometer No.1 at different temperature.

Volume of oil taken = 100ml

Volume of water taken = 100ml

S. No.	Temperature (°C)	Time for water (sec.)	Time for oil (sec.)
1.	At room temperature		
2.	40		
3.	50		
4.	60		

Calculations:-

Viscosity of diesel oil can be calculated as:

$$\frac{\eta_{oil}}{\eta_{water}} = \frac{t_{oil}}{t_{water}} \times \frac{d_{water}}{d_{oil}}$$

1. At room temperature
2. At 40°C
3. At 50°C
4. At 60°C

Result:-

Relative viscosity of light oil(diesel oil) at room temperature = -----poise

Relative viscosity of light oil(diesel oil) at 40°C = -----poise

Relative viscosity of light oil(diesel oil) at 50°C = -----poise

Relative viscosity of light oil(diesel oil) at 60°C = -----poise

VIVA VOCE

Q1. Define viscosity and viscosity index. Name few additives used as viscosity index improver.

Ans. Viscosity is the property of liquid in which a layer of liquid offers resistance to the flow of another layer whereas viscosity index gives the change in viscosity with temperature.

Q2. What do you mean by “ All weather lubricants” ?

Ans. Those lubricants whose viscosity does not change much with rise or fall of temperature are called “All time weather Lubricants”.

Q3. What do you mean by the term oiliness of a lubricating oil ? How can it be increased ?

Ans. Oiliness is defined as the sticking property of lubricants on machine surface to reduce friction. It depends upon temperature and pressure. By increasing pressure or decreasing temperature we can increase oiliness.

Q4. What is the difference between Redwood viscometer No.1 & no.2 ?

Ans. The difference between Redwood viscometer No.1 & no.2 are -

- ❖ Viscometer No. 1 is used for less viscous lubricants while No. 2 is used for high viscous lubricants.
- ❖ They mainly differ in their orifice size. Redwood No. 1 is used for oils having $t = 30-2000 \text{ Sec}$ & Redwood No. 2 for $t \geq 2000 \text{ Sec}$

Q5. Define coefficient of viscosity and mention its units.

Ans. Co-efficient of viscosity is defined as the frictional force area, separated by a unit distance & the velocity difference between those two layers is 1 cm/sec & it is denoted by (η).

- ❖ Its unit is $\text{Kgm}^{-1} \text{sec}^{-1}$ or poise or dyne Sec m^{-2}

EXPERIMENT NO: -8

AIM:- To find out the concentration of given KMnO_4 solution spectrophotometrically.

Apparatus:- Spectrophotometer, 100ppm KMnO_4 solution in distilled water, Cuvettes, Beaker.

Theory:- Spectrometry involves analysis of chemicals through measurement of intensity of light absorbed or transmitted through the solution as function of wavelength.

Absorbance (A) or optical density (O.D.) is given by:-

$$A = \log I_0/I_t$$

Where I_0 = Intensity of incident radiations.

I_t = Intensity of radiations after passing through the solution (i.e. transmitted radiations)

From Beer Lambert's law

$$A = \epsilon ct$$

Where ϵ = Molar extinction coefficient or molar absorptivity

c = Concentration

t = Thickness of absorbing medium.

If the some spectrophotometer cell is used for measuring absorbance of the solutions of different concentrations. Then Beer Lambert's law equation reduces to:

$$A = \epsilon c \text{ or } A \propto c \text{ (as } t \text{ thickness is constant)}$$

Thus, if a graph is plotted between A & c, we get a straight line passing through the origin for the solution obeying Beer Lambert's law.

This graph between A & c is used as calibration curve or graph for finding unknown concentration of solution under examination.

Procedure:-

1. Prepare the standard stock solution (100ppm) of KMnO_4 by dissolving 0.1 gm KMnO_4 /L of Solution.
2. Then prepare 80ppm , 60ppm, 40ppm, 20ppm separately in volumetric flask by appropriate dilution of stock solution.
For example 8ml stock solution + 2ml distilled water = 80ppm and so on.
3. Take 20ppm solution and note down the wavelength value where maximum absorption takes place (wavelength at which instrument gives maximum absorbance is called λ_{max} (in this case 510 – 515nm).
4. Fix the wavelength knob at λ_{max} .
5. Keep the sensitivity knob at position 1.
6. Set the display to 100.0 % transmittance without inserting the cell by using set zero knobs.
7. Now open the lid and insert cell with blank solution. Close the lid and bring the display reading near to 100 % transmittance by calibrate coarse control and then bring the display to 100 % by calibrate fine control.
8. Now take absorbance of all the solutions from 100ppm to 10ppm one by one by inserting the cell with appropriate concentration solution and reading from display.
9. Draw a calibration curve by plotting A vs. c. (it should be straight line passing through the origin).

10. Take absorbance of known solution and find the conc. curve from the calibration graph.

Precautions:-

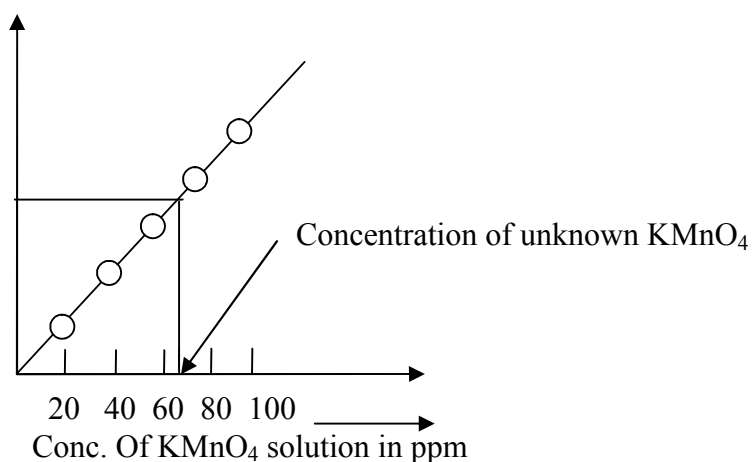
1. Always use dilute solution for getting calibration curve.
2. Cuvette should be cleaned properly and must be wiped with tissue paper.
3. Do not leave any finger mark on the cuvette.

Observation table:-

Concentration of KMnO_4 solution versus absorbance at $\lambda_{\text{max}} = 515\text{nm}$

S.No.	Concentration (KMnO_4 solution)	Absorbance at λ_{max}
1.	20ppm	
2.	40ppm	
3.	60ppm	
4.	80ppm	
5.	100ppm	
6.	Unknown conc.	

Calibration curve:-



Concentration of unknown KMnO_4 solution observed from calibration curve = -----ppm

Result:- Concentration of unknown KMnO_4 solution = -----ppm

VIVA VOCE

Q-1 what do you understand by calibration curve?

Ans. The absorbance is plotted against the concentration curve is called calibration curve.

Q-2 what is I-max? What is the significance of determining it.

Ans. I-max –intensity of absorption at max value for obtaining I-max: KMnO_4 solution .it helps in determining the intensity for KMnO_4 solution.

Q-3. What is the difference between calorimeter and a spectrophotometer?

Ans. Spectrophotometer involves the measurement of intensity of light absorbed by transmitted through solution.

Q-4. In calorimetric estimation of each metal in solution a particular filter is selected .give reason.

Ans. For different colour we have different value for intensity max .Each metal possesses a specific colour and therefore we require a particular filter.

Q-5. State “Lambert Beer” law and write the expression for it .

Ans. The intensity of incident light is proportional to the length of thickness absorbing material and concentration of solution

$$A = ECx$$

Q-6. Define molar extinction coefficient?

Ans. The reciprocal of thickness of 1M Solution which reduces the intensity of light to 1/10 of its value.

Q.7. How will you verify Lambert-Beer's Law?

Ans. If absorbance is plotted against conc. & the graph a straight line is obtained ,this verifies the Lambert-Beer's law.

Q.8. Given KMnO_4 solution obeys Beer's law justify the statement?

Ans. The graph b/w absorbance plotted against conc. Is straight line suggest that KMnO_4 solution obey Beer's law.

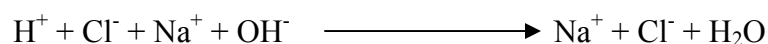
EXPERIMENT NO: - 9

AIM:- Determination of strength of HCl solution by titrating it against NaOH solution conductometrically.

Apparatus:- Conductometer, conductivity cell, beaker, burette, pipette, funnel etc.

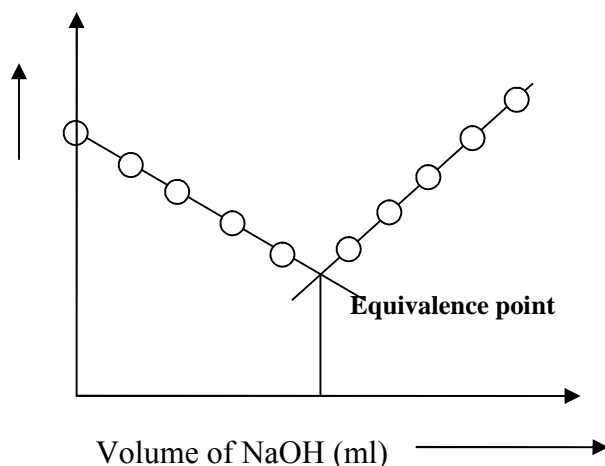
Chemicals:- 0.1N NaOH, standard conductivity reagent (KCL solution) and unknown HCl solution.

Theory:- Known volume of acid (say 50ml) is taken in conductivity vessel (beaker) and titrated it against standard NaOH solution. At each stage the conductance of reaction mixture is determined.



Graph is plotted as conductance vs volume of NaOH added, intersect of two of two lines gives equivalence point and volume of NaOH used is read out and then the strength is calculated.

Graph:-



Procedure:-

1. Rinse the conductivity cell with the solution whose conductivity is to be measured.
2. Take 50ml of the given HCl solution in a 100ml beaker.
3. Wash the conductivity cell with distilled water and then rinse it with the given HCl solution. Dip the cell in the solution taken in a beaker.
4. Connect the conductivity cell to the conductometer.
5. Set the switch to check position. Display must read 1.000. If it does not, set it with CAL control at the back panel.
6. Put the "function switch" to "cell constant" and set the value of cell constant at 1.000 with the help of cell constant knob.
7. Set the temperature control to the actual temperature of the solution under test.
8. Set the "function switch" to conductivity and read the display. This will be the exact conductivity note it down.

9. Take alkali (NaOH) in the burette and add 0.5ml of it in to the beaker containing HCl. Stir and determine the conductivity.
10. Repeat the procedure of addition of 0.5ml of NaOH and note down the conductivity in the Observation table. Take 12-15 readings in this way. After each addition stir the solution.
11. Plot the graph between conductance vs volume of NaOH added and find out the equivalence point and calculate the strength of HCl solution.

Precautions:-

1. The conductivity cell should be handled very carefully as it is very delicate.
2. The titrant solution should be 10 to 100 times stronger then the solution to be titrated so that there should be no appreciable change in the volume during the course of titration.
3. Stirring should be done after addition of titrant.

Observations:-

Volume of unknown HCl solution taken = $V_1 = 50.0\text{ml}$

Normality of NaOH = $N_2 = 0.1\text{N}$

Table

Titration between HCl and 0.1n NaOH solutions

S.NO.	Volume of NaOH added (ml)	Specific conductance (mho/cm)
1.	0.0	
2.	0.5	
3.	1.0	
4.	1.5	
5.	2.0	
6.	2.5	
7.	3.0	
8.	3.5	
9.	4.0	
10.	4.5	
11.	5.0	
12.	5.5	
13.	6.0	
14.	6.5	
15.	7.0	

Volume of NaOH used as found from the graph = $V_2 = \text{-----}$

Calculations:-

$$N_1V_1 = N_2V_2$$

$N_1 = \text{Normality of HCl} = ?$

$V_1 = \text{Volume of HCl} = 50.0\text{ml}$

$N_2 = \text{Normality of NaOH} = 0.1\text{N}$

$V_2 = \text{Volume of NaOH from graph} = \text{-----}$

Strength of HCl solution = Eq. weight X normality = $36.5 \times N_1$ g/l

Result:- Strength of given HCl solution as determined from conductometric titration is = -----g/l

VIVA VOCE

Q1. What are conductor metric titrations ? What is their main advantage over ordinary titrations ?

Ans. Conductor metric titrations measures the changes in ionic composition throughout chemical reaction in aqueous solution.

Q2. Give the factors on which conductance of a given solution depends ?

Ans. Conductance depends on two factors:-

1. No.s of ions
2. Mobility of ions

Q3. In the experiment you have done what is the PH of the solution before & after the titration ?

Ans. In metal the conductance is due to movement of free ions. Before titration solution contains HCL therefore PHS is acidic, after titration solution contains NaOH therefore PH is basic.

Q4. What is specific conductivity ? Give its units

Ans. Specific conductivity is defined as the conductance of ions per centimeter cube of solution.

- ❖ It is denoted by k { kappa }.
- ❖ Specific conductance = cell constant / absorbed resistance
- ❖ Its units are $\text{ohm}^{-1}\text{cm}^{-1}$ or mhocm^{-1} or scm^{-1}

Q5. What is the difference between conductance of metal and electrolytic solutions?

Ans. In metals the conductance is due to movements of free electrons. In electrolytic solution, the conductance is due to moving ions.

EXPERIMENT NO:- 10

AIM:- To determine the flash and fire point of a given sample of lubricating oil by Pensky - Marten's closed cup apparatus.

Requirements:- Pensky – Marten's closed cup apparatus, thermometer, lighter, beaker.

Chemicals:- Given sample of lubricating oil.

Theory:-

Flash and fire point:- The *flash point* of an oil is the lowest temperature at which it gives off vapours that will ignite for a moment when a small flame is brought near it.

The *fire point* of an oil is the lowest temperature at which the vapours of the oil burn continuously for at least 5 seconds when a small flame is brought near it.

The flash point and fire point are used to indicate the fire hazards of petroleum products and evaporation losses under high temperature operations. Knowledge of flash and fire points of lubricating oil aids in precautionary measures against fire hazards. A good lubricant should have flash point at least above the temperature at which it is to be used. Flash point is also used as a means of identification and also for detection of contamination of the lubricating oil.

Measurement of flash and fire points of a lubricating oil:-

The Pensky – Marten's closed cup method:- It is used to determine the flash point of lubricating oils, fuel oils, solvents, solvent containing materials and suspension of solids, except cut back asphalt. It gives more reproducible results.

It consists of a cup made of brass, which is about 5.5 cm deep and 5 cm in diameter. The lid of the cup is provided with four openings of standard sizes.

Through one of these openings passes a stirrer carrying two brass blades; while the second opening is meant for admission of air. Through the third opening passes a thermometer. While the fourth is meant for introducing test flame.

At the top of the cup, a shutter is provided. By moving the shutter, opening in the lid opens and flame (from flame exposure device) is dipped in to this opening, thereby bringing the flame over the oil surface. As the test – flame is introduced in the opening, it gets extinguished, but when the test flame is returned to its original position, it is automatically lighted by the pilot burner. There is also a stove consisting of an air bath and a top plate on which the flange of the cup rests. The air bath may be either a flame – heating metal casting or an electric resistance element. Pensky – Marten's flash point apparatus.

Procedure:-

- (a) The Cleveland cup is filled with oil sample to be tested up to the specified filling mark. There should be no oil on the outside of the cup. The thermometer is immersed in the sample which is then heated at a rate of 9° to 11° F per minute.
At every 5°F rise in temperature a small flame is passed over the oil surface. When a flash appears at any point on the surface of the oil, the temperature reading is recorded as the flash point. The heating of the oil is continued at the same rate. The test – flame is applied again for every 5° F rise in temperature until oil ignites and continues to burn for at least 5 seconds. The temperature reading is recorded as fire point. Fire point is generally from 10° to 70° F higher than the flash point.
- (b) The oil sample is filled up to the specified filling mark in the Pensky – Marten's cup. It is then

covered and positioned properly in the stove. The thermometer is then inserted in the sample. The test flame is lighted, while being stirred, the sample is heated at the rate of 9° to 11° F per minute. Up to 220° F, the test flame is applied at every 2° F rise in temperature, thereafter it is applied at every 5° F rise in temperature. When a distinct flash occurs in the interior of the cup at the time of the flame application, the temperature reading on the thermometer is the flash point.

The test flame is applied again for every 5°F rise in temperature until oil ignites and continuous to burn for at least 5 seconds. The temperature reading is recorded as the fire point.

Result:-

(1) The flash point of a given sample of lubricating oil = -----

(2) The fire point of a given sample of lubricating oil = -----

Precautions:-

- (a) Properly wash and dry the oil cup. There should be no moisture inside the cup.
- (b) Fill the supplied sample of a lubricating oil up to the specified filling mark. There should be no oil on outside the cup.
- (c) The thermometer should be immersed in the oil sample.
- (d) Avoid breathing over surface of oil.

VIVA VOCE

Ques: - What is flash point?

Ans:-It is the lowest temperature at which oil gives off vapours that will ignite for a moment when a small flame is brought near it.

Ques: - What is fire point?

Ans: - It is the lowest temperature at which vapours of oil burn continuously for at least 5 sec when a small flame is brought near it.

Ques:-What are functions of lubricating oil?

Ans: - It acts as cooling agent, sealing agent and corrosion preventer.

Ques:-How lubricating oil are classified?

Ans:-

1. Vegetable & animal Oil
2. Mineral Oil
3. Blended Oil

Ques: - Where flash and fire point is used?

Ans They is used to indicate fire hazards of petroleum products and evaporation losses under high temperature operations.

