# UNIT V

# **CORROSION ENGINEERING**

## **5.1 CORROSION**

## 5.1.1 Introduction

Corrosion is a 'billion dollar thief'. Even though it is a natural phenomenon in which the gases present in the atmosphere react chemically with metals to convert them into their salts, it results in loss of material and money. Metals have a strong crystalline structure and when they are converted into their salts they lose the metallic strength resulting in the damage to machineries in which they are used. Thus corrosion causes damage to metals and there by to the society. The estimate of loss due to corrosion is approximately 2.5 billion dollars / annum all over the world. Hence it is necessary to understand the mechanism of corrosion. In this lesson we are going to study about the causes and the mechanism of corrosion so that we can find ways to prevent this social enemy.

Corrosion is defined as the deterioration of a metal by chemical or electro chemical reactions with its environment.

Due to corrosion the useful properties of a metal like malleability, ductility, electrical conductivity and also the surface appearance are lost.

The most familiar example of corrosion is rusting of iron when exposed to atmospheric conditions.

Another example is the formation of green film or basic Carbonate  $[CuCO_3+Cu(OH)_2]$  on the surface of copper when exposed to moist air containing  $CO_2O_2$ 

#### 5.1.2 Types of Corrosion:

- 1. Chemical Corrosion or Dry Corrosion
- 2. Electro chemical Corrosion or Wet Corrosion

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## (1) Chemical Corrosion or Dry Corrosion

The direct chemical action of atmospheric gases like oxygen, halogen,  $H_2S$  etc in a dry environment on metals, a solid filim of the Corrosion produced is formed on the surface of the metal. This is known as chemical Corrosion.

A solid film of the corrosion product is formed on the surface of the metal which protects the metal from further corrosion. If a soluble or volatile corrosion product is formed, then the metal is exposed to further attack. 'For example, chlorine attack silver generating a protective film of silver chloride on the surface.

 $2 \text{ Ag+Cl}_2 \rightarrow 2 \text{AgCl}.$ 

## (2) Electro Chemical Corrosion or wet corrosion

Wet corrosion occurs due to the electrochemical action of moisture and oxygen on metals. Corrosion of very important metal, iron takes place due to electrochemical attack.

There are two theories proposed to explain rusting of iron.

- 1) Galvanic cell formation theory
- 2) Differential aeration theory (concentration cell theory)

## 5.1.3 Galvanic Cell Formation Theory

- When iron piece with impurity is exposed to atmosphere, a mini galvanic cell is formed.
- Iron with atmosphere forms one electrode.
- Impurity (copper, tin, dirt, etc) with atmosphere forms another electrode.

## \* Anodic reaction

Iron which is more electropositive acts as **anode**. Therefore iron is oxidized to ferrous ions ( $Fe^{2+}$  ions) by the removal of electrons. Ferrous ions combine with hydroxide ions to form ferrous hydroxide by atmosphere. Finally ferric hydroxide decomposes to form ferric oxide, which is nothing, but rust.

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

$$Fe^{2+} + 2OH^{-} \rightarrow Fe(OH)_{2}$$

$$[O]$$

$$Fe(OH)_{2} \rightarrow Fe(OH)_{3}$$

$$Fe(OH)_{3} \rightarrow Fe_{2} O_{3} + H_{2}O$$

The electrons released at the anode are absorbed at the cathode to form either hydrogen or water or hydroxide ion depending on the nature of the atmosphere.

This completes the formation of the cell, which favours rusting of iron.

## **Cathodic reaction**

Depending on the nature of the atmosphere the following reactions take place at the cathode.

When the atmosphere is acidic and contains no oxygen, hydrogen will be given out.

 $2H^++2e^-\rightarrow H_2$ 

When the atmosphere is acidic and contains more oxygen water will be given out.

 $2H^++[O]+2e^-\rightarrow H_2O$ 

When the atmosphere is basic or neutral and contains no oxygen,  $OH^2$  and  $H_2$  will be given out.

 $2 H_2O + 2 e^- \rightarrow H_2 + 2OH^-$ 

When the atmosphere is basic or neutral and contains more oxygen OH<sup>-</sup> will be given out.

## $2H_2O+O_2+4e^-\rightarrow 4OH^-$

# (2) Differential aeration theory or concentration cell formation theory

According to this theory, corrosion occurs due to the development of concentration cell formed by varying concentration of oxygen or any electrolyte on the surface of the metal.

Thus,

The less oxygenated area acts as Anode (gets corroded)

The more oxygenated area acts as the Cathode (Protected from Corrosion)



Reaction

At anode (less oxygenated area)  $Fe \rightarrow Fe^{2*} + 2e^{-}$  (oxidation or corrosion) At the cathode (more oxygenated area)

 $2 H_2 0 + O_2 + 4e^- \rightarrow 4OH^-$  (Reduction)

 $Fe^{2+} + 2OH \rightarrow Fe(OH)_2$ 

 $Fe(OH)_2$  is further oxidized to  $Fe(OH)_3$ . Since the anodic area is small and the cathodic area is large, corrosion is more concentrated at the anode. Thus, a small hole is formed on the surface of the metal. This type of intense local corrosion is called **pitting**.

Other examples for differential aeration corrosion are:

- 1. Corrosion noted on the barbed wire fencing, In a wire fence, the areas where the wires cross are less accessible to air than the rest of the fence and hence corrosion takes place at the wire crossings which are anodic.
- 2. Corrosion noted in the iron water tanks near the water level water line corrosion.



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In iron water tanks, iron portion inside the water is less expose to the oxygen when compared to other portions. Thus a concentration cell is formed and iron rusting takes place at the water level. (the place where the anode and cathode meet). The rust spreads when the water evaporates. This type of corrosion is called water line corrosion.

3. When a drop of water or salt solution is placed over an iron piece corrosion occur at the ridge of the water drop



Areas covered by droplets, having less access of oxygen become anodic with respect to the other areas which are freely exposed to air.

## 5.1.4 Factors affecting the rate of corrosion

The factors that affect he rate of corrosion are

- 1) Factors connected with the metal and its surface
- 2) Factors connected with the atmosphere
- 3) Factors connected with the corrosion product

## Factors connected with the metal:

## 1) Position of the metal in the E.M.F Series

The type of impurity present in it and its electropositive nature decides the corrosion of a metal. For example when iron has impurities like copper, tin, etc. iron corrodes since iron is more electropositive than metals like copper and tin. On the other hand when iron is coupled with zinc, zinc corrodes since zinc is more electropositive than iron.

## 2) Purity of the metal.

Generally pure metal does not corrode, as there is no cathode spot available to induce corrosion.

## 3) Surface of the metal

A rough surface corrodes readily as it collects more dirt and provides more cathode spot for corrosion. A polished surface does not corrode easily.

## 4) Stress corrosion

Stress in a metal surface is produced by mechanical workings such as quenching, pressing, bending, and riveting, improper heat treatment etc. The portion subjected to more stress acts as anode and other portion act as cathode. This leads to the formation of stress corrosion. Stress corrosion is noted in fabricated articles of certain alloys like high zincbrasses and nickel brasses.

Caustic embrittlement noted in boilers is a typical example for stress corrosion, which is due the attack of alkali present in water on stressed boiler metal.

#### 5) Anode to cathode area ratio.

When a bigger cathode area covers a smaller anode area, severe corrosion is noted in the anode spot. This is called erosion. It is frequently encountered in piping agitators, condenser tubes etc. where turbulent flow of gases and vapors remove the coated surfaces resulting in differential cells. Removal of surface coatings can also be caused by rubbing or striking activities of solids on the coated surfaces.

## 6) Physical state of a metal.

The rate of corrosion is influenced by grain size, Orientation of crystals, stress etc. The smaller the grains size of the metal greater the rate of corrosion.

#### Factors connected with the atmosphere

- 1) Nature of the atmosphere
- 2) Temperature of the atmosphere
- 3) pH of the atmosphere
- 4) Amount of moisture in the atmosphere
- 5) Amount of oxygen in the atmosphere
- 6) Amount of chemical fumes in the atmosphere etc.

## Examples

- 1. Buried pipelines and cables passing from one type of soil to another suffer corrosion due to differential aeration.
- 2. Lead pipe lines passing through clay and then through sand.
- 3. Lead pipe line passing through clay get corroded because it is less aerated than sand.

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## Factors connected with the corrosion product

a. In some causes the corroded product sticks to the surface and absorbs more moisture. This induces further corrosion.

E g Rusting of iron, As rust formed over iron absorbs more moisture, rate of rusting of iron increases

b. In some cases the corroded product acts as the protective coating which prevents further corrosion.

E g Aluminium oxide formed over the surface of aluminium prevents further corrosion and act as a protective coating. This is the basic principle of anodization.

c. In some other cases the corroded product falls out of position exposing the fresh metal surface for further corrosion.

E g Magnesium Oxide formed over the surface of Magnesium falls out of position exposing a fresh surface for further corrosion.

#### SUMMARY

In this lesson various types of corrosion, theories explaining corrosion and factors influencing corrosion are explained.

## QUESTIONS:

## Part A

- 1. What is corrosion?
- 2. What is dry corrosion?
- 3. What is wet Corrosion?
- 4. What type of corrosion takes place in a metal when anode is small and cathode is large. Why?

## Part B

- 1. Explain the Galvanic cell formation theory.
- 2. Explain the differential aeration theory with suitable examples.
- 3. What are the factors affecting the rate of corrosion.

## **TEST YOUR UNDERSTANDING**

- 1. Why corrosion often takes place under metal washers.
- 2. Welded joints are better than riveted joints. Why?

# **5.2 METHODS OF PREVENTION OF CORROSION**

## 5.2.1 Modifying the Environmental Conditions.

The corrosion rate can be reduced by modifying the environment. The environment can be modified by the following:

- (a) Deaeration: The presence of increased amounts of oxygen is harmful since it increases the corrosion rate. Deaeration aims at the removal of dissolved oxygen. Disolved oxygen can be removed by deaeration or by adding some chemical substance like Na 2CO<sub>3</sub>.
- (b) Dehumidification: In this method, moisture from air is removed by lowering the relative humidity of surrounding air. This can be achieved by adding silica gel which can absorb moisture preferentially on its surface.
- (c) Inhibitors: In this method, some chemical substance known as inhibitors are added to the corrosive environment in small quantities. These inhibitors substantially reduce the rate of corrosion.

## 5.2.2 Alloying:

Both corrosion resistance and strength of many metals can be improved by alloying, e-g. Stainless steels containing chromium produce a coherent oxide film which protects the steel from further attack. The other non-corrosive alloys are German silver, Aluminium bronze, Nickel bronze, Duralumin etc

#### 5.2.3 Surface Coating:

Corrosion of metal surfaces is a common phenomenon. To protect a metal surface from corrosion, the contact between the metal and the corrosive environment is to be cut off. This is done by coating the surface of the metal with a continues, non-porous material, insert to the corrosive atmosphere. Such a coating is referred to as surface coating or protective coating. In addition to protective action, such coatings also give a decorative effect and reduce wear and tear.

## **Objectives of Coating Surfaces**

- 1. To prevent corrosion.
- 2. To enhance wear and scratch resistance.

- 3. To increase hardness
- 4. To insulate electrically
- 5. To insulate thermally
- 6. To impart decorative colour.

## 5.2.4 Metallic Coating:

Surfacing coatings made up of metals are known as metallic coatings. These coatings separate the base metal from the corrosive environment and also function as an effective barrier for the protection of base metals.

The metal which is coated upon is known as the base metal.

The metal applied as coating is referred to as coat metal.

The different methods used for metal coating are.

- 1. Hot dipping
  - (a) Galvanization
  - (b) Tinning
- 2. Metal spraying.
- 3. Cladding.
- 4. Cementation
  - (a) Sherardizing Cementation with Zinc powder is called Sherardizing.
  - (b) Chromizing Cementation with 55% Chromium powder & 45% Alumina is called chromizing
  - (c) Calorizing Cementaion with Aluminium and Alumina powder is called Calorizing

5. Electroplating or electrodeposition.

# 1.Hot dipping

In the process of hot dipping, the metal to be coated is dipped in the molten bath of the coating metal. Such hot dip coatings are generally non-uniform. The common examples of hot dip coatings are galvanizing and tinning.

(a) Galvanizing: The process of coating a layer of zinc on iron is called galvanizing. The iron article is first pickled with dilute sulphuric acid to remove traces of rust, dust, etc. at 60-90'c for about 15 -20 minutes. Then this metal is dipped in a molten zinc bath maintained at 430'c. The surface of the bath is covered with ammonium chloride flux to prevent oxide formation on the surface of molten zinc. The coated base metal is then passed through rollers to correct the thickness of the film.

It is used to protect roofing sheets, wires, pipes, tanks, nails, screws, etc.

## (b) Tinning:

The coating of tin on iron is called tin plating or tinning. In tinning, the base metal is first pickled with dilute sulphuric acid to remove surface impurities. Then it is passed through molten tin covered with zinc chloride flux. The tin coated article is passed through a series of rollers immersed in a palm oil bath to remove the excess tin. Tincoated utensils are used for storing foodstuffs, pickles, oils, etc.

Galvanizing is preferred to tinning because tin is cathodic to iron, whereas zinc is anodic to iron. So, if the protective layer of the tin coating has any cracks, iron will corrode. If the protective layer of the zinc coating has any cracks, iron being cathodic does not get corroded. The corrosion products fill up the cracks, thus preventing corrosion.

Galvanising	Tinning
1.A process of covering iron with a thin coat of 'Zinc' to prevent it from rusting.	A process of covering iron with a thin coat of 'tin' to prevent it from corrosion.
2.Zinc protects the iron sacrificially.(Zinc undergo corrosion)	Tin protects the base metal with out undergo any corrosion (non sacrificially)
3.Zinc continuously protects the base metal even if broken at some places.	A break in coating causes rapid corrosion of base metal.
4.Galvanized containers cannot be used for strong acidic food stuffs as Zinc becomes toxic in acidic medium.	Tin is non-toxic in nature of any medium.

## 5.2.5 Differences between Galvanizing and Tinning.

## 5.2.6 Electroplating:

Electroplating is process in which the coat metal is deposited on the base metal by passing a direct current through an electrolytic solution.

## **Objectives of Electroplating:**

- a. To increase corrosion resistance.
- b. To get better appearance.
- c. To get increased hardness.
- d. To change the surface properties of metals and non metals.

## Process

In electroplating, the cleaned base metal is made as the cathode and the coat metal is taken as the anode. A solution of the coat metal salt is taken as the electrolyte. The electrodes are connected to a battery and DC current passed. Now electrolysis takes place and the coat metal deposited over the base metal.



The nature of coating depends on 1) the current density 2) time 3) temperature and 4) the concentration of the electrolyte.

For example, to coat silver on copper material, the copper material is taken as the cathode. A silver plate is taken as the anode. Silver thiocyanate solution is the electrolyte. When the electrodes are connected to a DC source of electricity, silver is deposited over the copper material.

The following electrolytes are used for coating other metals. Copper sulphate – Copper Nickel sulphate – Nickel Chromic acid – Chromium

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## Factors affecting electroplating

The following are the factors affecting electroplating:

- 1. Cleaning of the article is essential for a strong adherent electroplating.
- 2. Concentration of the electrolyte is a major factor in electroplating.
- 3. Low concentration of metal ions will produce uniform coherent metal deposition.
- 4. Thickness of the deposit should be minimized in order to get a strong adherent coating.
- 5. Additives such as glue and boric acid should be added to the electrolytic bath to get a strong adherent and smooth coating.
- 6. The electrolyte selected should be highly soluble and should not undergo any chemical reaction.
- 7. The pH of the electrolytic bath must be properly maintained to get the deposition effectively.

## 5.2.7 Anodizing:

Anodizing is the process of coating the base metal with an oxide layer of the base metal.

This type of coating is produced on metals like AI, Zn, Mg and their alloys by anodic oxidation process, by passing direct electric current though a bath in which the metal is suspended from anode. Here the base metal behaves as an anode. The electrolytes are sulphonic, chromic, phosphonic, oxalic or boric acid.

Anodised coating have more corrosion resistance due to thicker coating.

'Aluminium oxide coatings" are formed by the oxidation taking place on the aluminium surface at moderate temperatures (35 to 40°c) and moderate current densities. The formed oxide film is initially thin and gain thickness by the continous oxidation of aluminium anode. The surface of oxide film contains pores, which may cause corrosion. The pores can be sealed by exposing to boiling water, when the oxide is converted into monohydrate (Al<sub>2</sub>O<sub>3</sub>.H<sub>2</sub>O). This process is called sealing process.

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## 5.2.8 Phosphating:

Phosphate coatings are produced by the reaction between base metal and aqueous solution of phosphoric acid with accelerators (copper salt). Accelerators are used to enhance the rate of the reaction.

Such coatings are applied on iron, steel, zinc, aluminium, cadmium and tin. After the reaction the surface film consists of crystalline zinc iron or manganese iron phosphates.

Phosphate coatings do not prevent corrosion completely, they are principally used as an adherent base primer-coat for paint, lacquers, oils etc.

#### 5.2.9 Cathodic Protection:

The principle involved in cathodic protection is to force the metal behave like a cathode. Since there will not be any anodic area on the metal, corrosion does not occur. There are two types of cathodic protection.

(a) Sacrificial anodic method.

(b) Impressed voltage method.

#### (a) Sacrificial Anodic Method:

In this technique, a more active metal is connected to the metal structure to be protected so that all the corrosion is concentrated at the more active metal and thus saving the metal structure from corrosion. This method is used for the protection of sea going vessels such as ships and boats. Sheets of zinc or magnesium are hung around the hull of the ship. Zinc and magnesium being anodic to iron get corroded. Since they are sacrificed in the process of saving iron (anode), they are called

sacrificial anodes. The corroded sacrificial anode is replaced by a fresh one, when consumed completely.

Important applications of sacrificial anodic protection are as follows:

- (I) Protection from soil corrosion of underground cables and pipelines.
- (ii) Magnesium sheets are inserted into domestic water boilers to prevent the formation of rust.

## (b) Impressed voltage Method.

In this method, an impressed current is applied in an opposite direction to nullify the corrosion current and converting the corroding metal from anode to cathode. This can be accomplished by applying sufficient amount of direct current from a battery to an anode buried in the soil and connected to the corroding metal structure which is to be protected. The anode is in a backfill (composed of gypsum) so as to increase the electrical contact with the soil. Since in this method current from an external source in impressed on the system, this is called impressed current method.



Buried pipe made cathode

(protected)

This type of protection is given in the case of buried structures such as tanks and pipelines.

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## QUESTIONS

## Part - A

- 1. what is galvanization?
- 2. What is anodizing?
- 3. What is phosphating?
- 4. What is a base metal?
- 5. What is a coat metal?
- 6. Galvanization is preferred to tinning? Why?
- 7. What is Sherardizing?

## Part - B

- 1. Differentiate between galvanizing and tinning.
- 2. What are the factors that affect electroplating?
- 3. What is anodizing.? How is it carried out?
- 4. What is tinning?. What are its merits & demerits?
- 5. Write short notes on cathodic protection.

## TEST YOUR UNDERSTANDING

- 1. Why is moderate current density employed during electroplating?
- 2. Chromium anode are not used in chromium plating. Give Reason.

## **5.3 ORGANIC COATINGS**

## 5.3.1 Introduction

Organic coatings include paints and varnishes. In the this lesson we are going to study about paint and its components A little introductions to special paints used also discussed. further we are going to study about varnish, its types and their preparation .A preliminary idea is also given about dyes.

#### 5.3.2 Paint

Paint is a dispersion of a pigment in medium oil (vehicle). When paint is applied on a surface, the medium oil saves the surface from corrosion. The pigment saves the medium oil from the ultra violet light given by the sun.

#### 5.3.3 Components of paints and their functions

The important constituents of paint are as follows.

- 1. Pigment
- 2. Drying oil or medium oil or vehicle
- 3. Thinner
- 4. Drier
- 5. Filler or extender
- 6. Plasticizer
- 7. Antiskinning agent

## 1. Pigment:

A pigment is a solid and colour-producing substance in the paint.

#### Functions:

The following are the functions of the pigment:

- (a) A pigment gives opacity and colour to the film.
- (b) A pigment gives strength to the film.
- (c) It protects the film by reflecting the destructive ultraviolet rays.
- (d) It covers the manufacturing defects

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## 2. Drying oil or medium oil or vehicle.

The liquid portion of the which the pigment is dispersed is called a medium or vehicle. E.g. linsed oil, dehydrated castor oil, soyabean oil and fish oil.

#### Functions:

- (a) Vehicles hold the pigment particles together on the surface.
- (b) They form the protective film by evaporation or by oxidation and polymerization of the unsaturated constituents of the oil.
- (c) Vehicles give better adhesion to the surface.
- (d) They impart water repellency, durability and toughness to the film.

## 4. Thinner:

Thinners are added to paints to reduce the viscosity of the paints so that they can be easily applied to the surface. E.g. turpentine and petroleum sprit.

## Functions:

- (a) Thinners reduce the viscosity of the paint to render it easy to handle and apply to the surface.
- (b) They dissolve the oil, pigments etc. and produce a homogeneous mixture.
- (c) Thinners evaporate rapidly and help the drying of the film.
- (d) They increase the elasticity of the film.
- (e) Thinners increase the penetrating power of the vehicle.

## 4. Drier:

Driers are used to accelerate the drying of the oil film.

Eg Naphthenates of lead and cobalt, Resonates of lead and cobalt.

#### Functions:

Driers act as oxygen carrier catalysts which help the absorption of oxygen and catalyze the drying of the oil film by oxidation, polymerization and condensation.

## 5. Filler or extender:

Fillers are added to reduce the cost and increase the durability of the paint, E.g. talc, Gypsum, mica, asbestos etc.

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## Functions:

- (a) Fillers serve to fill the voids in the film.
- (b) They reduce the cracking of the paints.
- (c) Fillers increase the durability of the paints.
- (d) They reduce the cost of the paint.

## 6. Plasticizers:

Plasticizers are chemicals added to paints to give elasticity to the film and to prevent cracking of the film, e.g. triphenyl phosphate, tertiary amyl alchol, Tributyl Phthalate.

## 7. Antiskinning agent:

They are chemicals added to the paint to prevent skinning of the paint, E.g. polyhydroxy phenols.

## 5.3.4Varnishes

Varnish is a homogenous colloidal dispersion of natural or synthetic resin in oil or thinner or both. It is used as a protective and decorative coating to surfaces. It provides a hard, transparent, glossy, lustrous and durable film to the coated surface.

#### There are two types of varnishes

- 1. Oil Varnish
- 2. Spirit Varnish

## Preparation of oil Varnish

The resin is taken in an aluminium vessel, known as a kettle, and heated The resin melts and the temperature is slowly increased to about 300°C. This process, is known as gum running, Some cracking or depolymerization of the resin takes place and about 25 per cent of the resin is lost in the form of pungent fumes. The required quantity (about 25 per cent of the weight of the resin) of boiled oil or linsed oil along with driers is separately heated to 200 to 220 and is slowly added to the heated resin with constant stirring until thorough combination has taken place. This operation is known as cooking. The kettle is removed from the furnace and allowed to cool, white spirit, which is a thinner is added to varnish when the temperature of the varnish is below the flash point of the thinner with constant stirring during the addition.

The varnish is stored in tanks for some days for maturing. Filtered or packed for marketing.

## Preparation of sprit varnish

Sprit varnish is obtained by dissolving a resin in a sprit. E.g. shellac resin dissolved in methylated sprit, a solution of shellac in alcohol, etc.

Resins of trees like Manila, Damar, etc are also used for making varnish.

SI. No	Paint	Varnish
1.	Paint has pigment	There is no pigment in the varnish
2.	It can be applied to both metals & wooden articles	It can be applied only to the wooden articles
3.	It is Opaque	It is transparent

## Difference between paint and varnish

#### 5.3.5 Special paints

In addition to the normal ingredients some special chemicals are incorporated to paints for some specific purposes. They are commonly known as special paints.

#### 1. Luminescent paints

Luminescent paints contains luminophor pigments are used for visibility in the dark. They find application in inks, advertising signboards, road marks, number plates of vehicles, watch dials, etc. The active components in luminous paint are specially prepared phosphorescent materials like CaS, ZnS, etc. They absorb light radiations and emit them in the dark. For colour effect in luminous paints, certain chemicals like copper salts (green), silver salts (blue), cerium and uranium salts (yellow), etc. are used.

## 2. Heat Resistance paints

When the surfaces are exposed to high temperatures such as in chimneys, exhaust pipes, furnaces, oil stills, etc. Oil paints tend to decompose or get charred, they being organic in nature. Then the surfaces become liable for corrosion. To overcome this problem, a suspension of graphite or lamp black in small amounts of drying oils and more thinners can be used. But more recently, silicone paints are used for heat resistance.

## 3. Fire-retardant paints

These are paints containing chemicals which are fire-resistant in nature. In other words, they produce gases like  $CO_2$ ,  $NH_3$ , HCl,HBr on heating which are themselves non-combustible and do not support combustion, there by minimizing the rate of burning or extinguishing the fire.

#### 4. Antifouling paints

Oil paints are liable for attack by living organisms because of the organic content in them. So, in places where living organisms are handled or are present, such paints cannot be used. For use in breweries and biochemical laboratories, the paint is mixed with compounds having fungicidal properties. The active ingredients employed are HgO,  $Cu_2O$ ,  $Hg_2CI_2$ , DDT, pentachlorophenol, etc.

Such paints are called Antifouling paints.

## 5. Cement Paint

Cement paint is the coating, which is applied on plastered brickwork, concrete work, etc. The ingredients are

- 1. White cement (about 70%)
- 2. Hydrated lime  $[Ca(OH)_2]$
- 3. Pigment (a colouring agent)
- 4. Very fine sand (an inert filler) and
- 5. Water-repellent compound

Such paints of different colors are marked in powder form (eg Snowcem, Smocem). The powder is mixed with a suitable quantity of water to get a thin slurry, and applied on surfaces. For good results, a 1.5% to 2% aqueous solution of sodium silicate and Zinc sulphate is applied as primer coat.

## 6. Aluminium Paint

The base material in aluminium paint is a fine powder of aluminium. The finely ground powder of aluminium is suspended in either spirit varnish or in an oil-varnish depending on the requirement. When paints is applied, the thinner evaporates and oil, if any, undergoes oxidation and polymerization. A bright adhering film of aluminium is obtained on the painted surface.

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## Advantages of aluminium paint:

- 1. It possesses a good covering power.
- 2. It imparts very attractive appearance to the surface.
- 3. It has fairly good heat-resistance.
- 4. It has very good electrical resistance
- 5. The painted film is waterproof.
- 6. The electrical surface is visible even darkness.
- 7. Corrosion protection for iron and steel surface is better than all other paints.

## 7. Distempers

Distempers are water paints. The ingredients of distemper are

- 1. Whitening agent or chalk powder (the base)
- 2. Glue or casein (the binder)
- 3. Colouring pigment and
- 4. Water (the solvent or thinner).

## Advantages

- 1. Distempers are cheaper than paints and varnishes
- 2. They can be applied easily on plasters and wall surfaces in the interior of the buildings.
- 3. They are durable.
- 4. They give smooth and pleasing finish to walls.
- 5. They have good covering power.

## 5.3.6DYES

Dyes are coloured organic compounds capable of colouring various materials and articles.

According to commercial classification of dyes which is based on the application of the dyes are as follows.

- 1. Direct dyes
- 2. Basic dyes
- 3. Mordant dyes etc.

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## Acid Dyes:

Salts of organic acids are called acid dyes. It donate colour cellulose fibres. They can readily dye only animal fibres.

Eg: Methyl red, Methyl Organe etc.

## Basic Dyes.

They are salts of colour bases with hydrochloric acid or Zinc chloride. Basic dyes can dye animal fibres directly and vegetable fibres after the fibres are mordanted with tannin. Basic dyes are mostly used for dyeing silk and cotton.

Eg: Magenta, Para-rosaniline dye, Aniline Yellow.

## Mordant or Adjective dyes:

A mordant is any substance, which is fixed to the fibre before dyeing. Commonly used mordants are hydroxides or basic salts of chromium, aluminium or iron. Tannic acid is also used as mordant for basic dyes.

Generally the fabric is dipped in a mordant and then in a solution of dye. The dye coat thus formed is insoluble and does not fade on washing.

E.g. Alizarin and Anthraquinone dyes.

## Questions

## Part - A

- 1. Define paint.
- 2. Give examples for heat resistant paint.
- 3. What is Varnish?
- 4. What is the function of the drier in paint?
- 5. What are Fire-retardant paints?
- 6. Define Dyes.

## Part - B

- 1. What are the components present in the paint. Explain their functions.
- 2. How is oil varnish prepared?
- 3. Write a short note on special paints.
- 4. write short notes on Dyes.

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## **TEST YOUR UNDERSTANDING**

- 1. What are toners?
- 2. What is an enamel?

# SEMESTER-I PRACTICAL-I VOLUMETRIC ANALYSIS

The method to determine the exact amount of the substance in a given sample is termed as quantitative analysis volumetric analysis is a branch of quantitative analysis involving accurate measurement of volumes of reacting solutions. The volumetric analysis is very much in use due to simplicity rapidity accuracy and wide applicability.

The reacting substances are taken in the form of solutions and made to react. The concentration of one solution is determined using another suitable solution whose concentration is accurately known. A known volume of one solution is measured with a pipette and taken in a conical flask. The other solution is taken in a burette and run into the first solution till the chemical reaction is just complete. The volume of the second solution is read from the burette and the two volumes are compared.

#### Various terms used in volumetric analysis are given below:

#### Titration

The process of adding one solution from the burette to another in the conical flask in order to complete the chemical reaction is termed titration.

#### Endpoint

It is the exact stage at which chemical reaction involved in the titration is just complete

#### Indicator

It is a substance which will show the end point of the reaction by change of colour. For example phenolphthalein and methyl orange are indicators used in acid alkali titrations. Potassium permanganate itself acts as an indicator in potassium permanganate titrations.

#### Acidimetry and Alkalimetry Titration:

Acidimentry refers to the titration of alkali with a standard acid and alkalimetry refers to the titration of an acid with a standard alkali.

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## Permanganimetry Titration:

The titration involving  $KMnO_4$  is called permanganimetry titration. In presence of dilute  $H_2SO_4$   $KMnO_4$  oxidizes ferrous sulphate to ferric sulphate and oxidizes oxalic acid to  $CO_2$  and  $H_2O$ .

## Normality:

The strength of a solution is expressed in terms of normality. Normality is the number gram equivalent mass of solute dissolved in one litre of solution.

## Standard solution:

A solution of known strength (Normality) is called a standard solution.

## **Decinormal Solution:**

A solution having the strength (Normality) of 0.1 N is called decinormal solution.

## Law of volumetric analysis:

Whenever two Substances react together, they react in the ratio of their equivalent mass. One litre of a normal solution of a substance will react exactly with same volume of a normal solution of another substance. In other words equal volumes of equal normal solutions will exactly react with each other. This result is stated in the form law of volumetric analysis

If  $V_1$  ml of a solution of strength  $N_1$  is required or complete reaction by  $V_2$  ml of the second solution of strength  $N_2$  then

$$V_1N_1 = V_2N_2$$

If any three factors  $(V_1V_2 \& N_1)$  are known, the fourth factor  $N_2$  can be calculated. The following are the important formula used in all volumetric estimations

Mass of solute per litre of the solution = Equivalent mass x Normality

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Name of the compound	Equulvalent Mass
Hydrochloric acid	36.5
Sulphuric acid	49
Oxalic acid	63
Sodium carbonate	53
Sodium hydroxide	40
Potassium hydroxide	56.1
Potassium permanganate	31.6
Ferrous sulphate	278
Ferrous ammonium sulphate	392
Potassium dichromate	49.04
Copper sulphate	249.54
EDTA (disodium salt)	372

## Equivalent mass of some important compounds

# **1 ESTIMATION OF SULPHURIC ACID**

# EX.NO

## Date.....

#### Aim

To estimate the amount of Sulphuric acid present in 400 ml of the given solution. You are provided with a standard solution of oxalic acid of normality .....N and an approximately decinormal solution of Sodium hydroxide. (Test solution should be made up to 100 ml)

#### Principle

The titration is based on the neutralisation reaction between oxalic acid and Sodium hydroxide in titration I and Sulphuric acid and Sodium hydroxide in titration II.

#### Procedure

#### Titration I:

## Standardisation of Sodium hydroxide

The burette is washed with water, rinsed with distilled water and then with the given oxalic acid. It is filled with same acid up to zero mark. The initial reading of the burette is noted. A 20ml pipette is washed with water, rinsed with distilled water and then with the given Sodium hydroxide solution.20 ml of Sodium hydroxide is pipetted out in to a clean conical flask. Two drops of phenolphthalein indicator is added into the flask. The solution becomes pink in colour. The solution is titrated against oxalic acid taken in the burette. The end point of the titration is the disappearance of pink colour to give colourless solution. The titration is repeated to get the concordant value. From the titre value, the normality of Sodium hydroxide is calculated.

#### Titration II:

## Standardisation of Sulphuric acid

The given Sulphuric acid solution is made upto 100 ml in a 100 ml standard flask. The solution is thoroughly shaken to get a uniformly concentrated solution. The burette is washed with water, rinsed with distilled water and then with the given Sulphuric acid from the standard

flask. It is filled with same acid upto zero mark. The initial reading of the burette is noted. Exactly 20 ml of the sodium hydroxide is pipetted out in to a clean conical flask. To this solution two drops of phenolphthalein indicator is added. The solution becomes pink in colour. The solution is titrated against Sulphuric acid taken in the burette. The end point of the titration is the disappearance of pink colour to give colourless solution. The titration is repeated to get the concordant value. From the titre value, the normality of Sulphuric acid and the amount of sulphuric acid present in 400 ml of the given solution is calculated.

#### Result

Normality of Sodium hydroxide Normality of Sulphuric acid Amount of Sulphuric acid present in 400ml of the given solution

=	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	Ν	l
=																											Ν	l

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#### Titration-I: Sodium hydroxide Vs Oxalic acid

S.No	Volume of sodium	Bure readin	ette g (ml)	Volume of oxalic	Indicator		
	hydroxide(ml)	initial	final	acid(ml)			

#### Concordant value=

#### Calculation:

- Volume of oxalic acid $(V_1) =$ Normality of oxalic acid $(N_1) =$ Volume of sodium hydroxide $(V_2) = 20 \text{ mI}$
- Normality of sodium hydroxide  $(N_2) =$

By the principle of volumetric analysis,  $V_1N_1 = V_2N_2$ 

$$\therefore N_2 = \frac{V_1 N_1}{V_2} = \frac{X}{20} = ----N$$

 $\therefore$  Normality of sodium hydroxide (N<sub>2</sub>) = \_\_\_\_\_ N

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S.No	Volume of sodium	Bure reading	ette g (ml)	Volume of sulphuric	Indicator			
	hydroxide(ml)	initial	final	acid(ml)				

## Titration-II: Sulphuric acid Vs Sodium hydroxide

#### Concordant value=

## Calculation:

Volume of sulphuric acid  $(V_1) =$  $(N_1) =$ Normality of sulphuric acid  $(V_2) = 20ml$ Volume of sodium hydroxide Normality of sodium hydroxide  $(N_2) =$ By the principle of volumetric analysis,  $V_1N_1 = V_2N_2$ 

$$\therefore N_1 = \frac{V_2 N_2}{V_1} = \frac{x \ 20}{v_1} = ----N$$

 $\therefore$  Normality of sulphuric acid (N<sub>1</sub>)= N Amount of sulphuric acid present in 400 ml of the given solution

?

=Equivalent mass x Normality of sulphuric acid x 400 / 1000

=49x-----x400/1000

## SHORT PROCEDURE

Description	Titration I	Titration II					
Burette solution Pipette solution Reagents added Indicator End point	Oxalic acid Sodium hydroxide  Phenolphthalein Disappearance of pink colour	Sulphuric acid Sodium hydroxide  Phenolphthalein Disppearance of pink colour					
Equivalent mass of sulphuric acid = 49							

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# 2. ESTIMATION OF SODIUM HYDROXIDE

#### EX.NO.....

#### Date.....

#### Aim

To estimate the amount of Sodium hydroxide present in one litre of the given solution. You are provided with a standard solution of Sodium carbonate of normality .....N and an approximately decinormal solution of Sulphuric acid. (Test solution should be made upto 100 ml ) Principle

The titration is based on the neutralisation reaction between Sulphuric acid and Sodium carbonate in titration I and Sulphuric acid and Sodium hydroxide in titration II.

#### Procedure

#### Titration I:

#### Standardisation of Sulphuric acid

The burette is washed with water, rinsed with distilled water and then with the given Sulphuric acid. It is filled with same acid upto zero mark. The initial reading of the burette is noted. A 20ml pipette is washed with water, rinsed with distilled water and then with the given Sodium carbonate solution.20 ml of Sodium carbonate is pipetted out in to a clean conical flask. Two drops of methyl orange indicator is added into the flask.The solution becomes pale yellow in colour. The solution is titrated against Sulphuric acid taken in the burette. The end point of the titration is the change in colour from yellow to permanent pale pink. The titration is repeated to get the concordant value. From the titre value, the normality of Sulphuric acid is calculated.

#### Titration II:

#### Standardisation of Sodium hydroxide

The given Sodium hydroxide solution is made upto 100 ml in a 100 ml standard flask. The solution is thoroughly shaken to get a uniformly concentrated solution. A 20 ml pipette is washed with water, rinsed with distilled water and then with the given Sodium hydroxide. Using the rinsed pipette, exactly 20 ml of the made-up solution is transferred into a clean conical flask. To this solution two drops of methyl orange indicator is added. The solution becomes pale yellow in colour. The solution is titrated against Sulphuric acid taken in the burette. The end point of the titration is

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the change in colour from yellow to permanent pale pink. The titration is repeated to get the concordant value. From the titre value, the normality of Sodium hydroxide is calculated.

## Result

- (i) Normality of Sulphuric acid
- (ii) Normality of Sodium hydroxide
- (iii) Amount of Sodium hydroxide present in one litre of given solution

=		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•		ľ	V
=	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	ľ	1

=.....g

## Titration-I: Sodium carbonate Vs sulphuric acid

S.No	Volume of sodium	Bure readin	ette g (ml)	Volume of sulphuric	Indicator		
	carbonate(ml)	initial	final	acid(ml)			

## Concordant value=

## Calculation:

/olume of sulphuric acid	$(V_1) =$
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Normality of sulphuric acid $(N_1) = ?$ Volume of sodium carbonate $(V_2) = 20 \text{ml}$ 

Normality of sodium carbonate  $(V_2) =$ 

Normality of socium carbonate

By the principle of volumetric analysis,  $V_1 N_1 = V_2 N_2$ 

: Normality of sulphuric acid  $(N_1)$  = \_\_\_\_\_N

$$:: N_1 = \frac{V_2 N_2}{V_1} = \frac{X \, 20}{V_1} = ----N$$

## Titration-II : Sulphuric acid Vs Sodium hydroxide

Volume of sodium	Bure readin	ette g (ml)	Volume of sulphuric	Indicator			
hydroxide(ml)	initial	final	acid(ml)				
	Volume of sodium hydroxide(ml)	Volume of sodium reading hydroxide(ml)	Volume of sodium hydroxide(ml)Burette reading (ml)initialfinal	Volume of sodium hydroxide(ml)Burette reading (ml)Volume of sulphuric acid(ml)initialfinal			

## Concordant value=

## **Calculation:**

Volume of sulphuric acid	(V <sub>1</sub> ) =
Normality of sulphuric acid	(N <sub>1</sub> ) =
Volume of sodium hydroxide	$(V_2) = 20mI$
Normality of sodium hydroxide	$(N_2) = ?$
By the principle of volumetric and	alysis, $V_1 N_1 = V_2 N_2$
$\therefore N_2 = \frac{V_1 N_1}{V_2} = -$	$\frac{X}{20} =N$
Normality of Sodium hydroxide	e(N <sub>2</sub> )=N
Amount of Sodium hydroxide present	1
in One litre of the given solution	$\int$ =Equivalent mass x Normality
	of sodium hydroxide
	= 40 x

#### SHORT PROCEDURE

Description	Titration I	Titration II	
Burette solution Pipette solution Reagents added Indicator End point	Sulphuric acid Sodium carbonate  Methyl orange Appearance of permanent pink colour	Sulphuric acid Sodium hydroxide  Methyl orange Appearance of permanent pink colour	
Equivalent mass of sodium hydroxide=40			

= -----g

# 3. COMPARISON OF STRENGTHS OF TWO ACIDS

## EX.NO.....

#### Date.....

#### Aim

To compare the strengths of two hydrochloric acids solutions in bottles A and B and estimate the amount of hydrochloric acid present in 250 ml of the weaker solution. You are provided with a standard solution of sodium hydroxide of normality ......N.

#### Principle

The experiment is based on the neutralisation reaction between hydrochloric acid A and Sodium hydroxide in titration I and hydrochloric acid B and Sodium hydroxide in titration II.

#### Procedure

#### Titration I:

#### Standardisation of hydrochloric acid A

The burette is washed with water, rinsed with distilled water and then with the given hydrochloric acid in bottle A. It is filled with same acid upto zero mark. The initial reading of the burette is noted. A 20ml pipette is washed with water, rinsed with distilled water and then with the given Sodium hydroxide solution.20 ml of Sodium hydroxide is pipetted out in to a clean conical flask. Two drops of phenolphthalein indicator is added into the flask. The solution becomes pink in colour. The solution is titrated against hydrochloric acid A taken in the burette. The end point of the titration is the disappearance of pink colour to give colourless solution. The titration is repeated to get the concordant value. From the titre value, the normality of hydrochloric acid A is calculated.

#### Titration II:

#### Standardisation of hydrochloric acid B

The burette is washed with water, rinsed with distilled water and then with the given hydrochloric acid in bottle B. It is filled with same acid upto zero mark. The initial reading of the burette is noted.20 ml of standardised Sodium hydroxide is pipetted out in to a clean conical flask. Two drops of phenolphthalein indicator is added into the flask. The solution becomes pink in colour. The solution is titrated against hydrochloric acid B taken in the burette. The end point of the titration is the disappearance of pink

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A =.....N B =.....N

 $(V_1) =$ 

 $(N_2) =$ 

 $(N_1) = ?$  $(V_2) = 20 \text{ ml}$ 

 $V_1N_1 = V_2N_2$ 

colour to give colourless solution. The titration is repeated to get the concordant value. From the titre value, the normality of hydrochloric acid B is calculated

#### Result

- 1. Normality of hydrochloric acid
- 2. Normality of hydrochloric acid
- Hydrochloric acid in bottle-----is weaker then hydrochloric acid in bottle------
- 4. Amount of hydrochloric acid present in 250 ml of the weaker solution =......g

## Titration-I: Sodium hydroxide Vs Hydrochloric acid A

S.No	Volume of sodium hydroxide(ml)	Burette reading (ml)		Volume of Hydrochlolic	Indicator
		initial	final	acid(ml)	

## Concordant value=

## Calculation:

Volume of hydrochloric acid A Normality of hydrochloric acid A Volume of sodium hydroxide Normality of sodium hydroxide By the principle of volumetric analysis,

$$:: N_1 = \frac{V_2 N_2}{V_1} = \frac{X20}{V_1} = ----N$$

 $\therefore$  Normality of Hydrochloric acid A = .....N

## Titration-II : Hydrochloric acid B Vs Sodium hydroxide

S.No	Volume of sodium hydroxide(ml)	Burette reading (ml)		Volume of Hydrochlolic	Indicator
		initial	final	acid(ml)	

## Concordant value=

## Calculation:

Volume of hydrochloric acid B	(V <sub>1</sub> ) =
Normality of hydrochloric acid B	$(N_1) = ?$
Volume of sodium hydroxide	$(V_2) = 20  \text{ml}$
Normality of sodium hydroxide	$(N_2) =$
By the principle of volumetric analysis,	$V_1 N_1 = V_2 N_2$
$:: N_1 = \frac{V_2 N_2}{V_1} = \frac{X20}{V_1} =$	— — N

...Normality of Hydrochloric acid B=.....N Hydrochloric acid in bottle.----is weaker than hydrochloric acid in bottle------Amount of hydrochloric acid present  $\int = Equivalent mass of HCI x$  $\int$  Normality of HCI x 250/1000

In 250 ml of the weaker solution

=.....g

## SHORT PROCEDURE

Description	Titration I	Titration II		
Burette solution Pipette solution Reagents added Indicator	Hydrochloric acid A Sodium hydroxide 	Hydrochloric acid B Sodium hydroxide  Phenolohthalein		
End point	Disappearance of pink colour	Disappearance of pink colour		
Equivalent mass of hydrochloric acid = 36.5				

# 4. COMPARISON OF STRENGTHS OF TWO BASES

## EX.NO.....

Date.....

#### Aim

To compare the normalities of Sodium hydroxide solutions supplied in bottles A and B and to estimate the amount of Sodium hydroxide present in 500 ml of the stronger solution. You are provided with a standard solution of Oxalic acid of normality .....N.

#### Principle

The titration is based on the neutralisation reaction between Oxalic acid and Sodium hydroxide.

#### Procedure

#### Titration I:

## Standardisation of Sodium hydroxide (A)

The burette is washed with water, rinsed with distilled water and then with the given oxalic acid. It is filled with same acid upto zero mark. The initial reading of the burette is noted. A 20ml pipette is washed with water, ,rinsed with distilled water and then with the given Sodium hydroxide solution in bottle A.20 ml of Sodium hydroxide A is pipetted out in to a clean conical flask. Two drops of phenolphthalein indicator is added into the flask. The solution becomes pink in colour. The solution is titrated against Oxalic acid taken in the burette. The end point of the titration is the disappearance of pink colour to give colourless solution. The titration is repeated to get the concordant value. From the titre value and normality of Oxalic acid. The normality of Sodium hydroxide in bottle A is calculated.

#### Titration II:

## Standardisation of Sodium hydroxide (B)

20 ml of Sodium hydroxide from bottle B is pipette out into a clean conical flask using clean rinsed pipette. To this solution two drops of phenolphthalein indicator is added. The solution becomes pink in colour. The solution is titrated against Oxalic acid taken in the burette. The end point of the titration is the disappearance of pink colour to give colourless solution. The titration is repeated to get the concordant value. From the titre value, the normality of Sodium hydroxide(B) is calculated.

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The two normalities are compared and the amount of Sodium hydroxide in 500ml of the stronger solution is calculated.

## Result

Normality of Sodium hydroxide in bottleA	=N
Normality of Sodium hydroxide in bottleB	=N
Sodium hydroxide in bottle	
is stronger then Sodium hydroxide in bottle	
Amount of Sodium hydroxide present in	
500ml of the stronger solution	=g

## Titration-I: Sodium hydroxide (A) vs Oxalic acid

S.No	Volume of sodium hydroxide A (ml)	Burette reading (ml)		Volume of oxalic	Indicator
		initial	final	acid(ml)	

## Concordant value=

## Calculation:

Volume of oxalic acid	$(V_1) =$
Normality of oxalic acid	$(N_1) =$
Volume of sodium hydroxide(A)	$(V_2) = 20  \text{ml}$
Normality of sodium hydroxide(A)	$(N_2) = 2$
By the principle of volumetric analysis,	$V_1 N_1 = V_2 N_2$
V N Y	

$$\therefore N_2 = \frac{V_1 N_1}{V_2} = \frac{X}{20} = ----N$$

 $\therefore$  Normality of Sodium hydroxide(A) (N<sub>2</sub>) = \_\_\_\_

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#### Titration-II : Sodium hydroxideB Vs Oxalic acid

S.No	Volume of sodium hydroxide B(ml)	Burette reading (ml)		Volume of oxalic	Indicator
		initial	final	acid(ml)	

#### Concordant value=



#### SHORT PROCEDURE

Description	Titration I	Titration II	
Burette solution Pipette solution Reagents added	Oxalic acid Sodium hydroxideA 	Oxalic acid Sodium hydroxideB 	
Indicator End point	Phenolphthalein Disappearance of pink colour	phenolphthalein Disappearance of pink colour	
Equivalent mass of sodium hydroxide = 40			

# 5. ESTIMATION OF MOHR'S SALT

#### EX.NO.....

#### Date.....

#### Aim

To estimate the amount of crystalline ferrous ammonium sulphate present in 100 ml of the given solution. You are provided with a standard solution of crystalline ferrous sulphate of normality .....N and an approximately decinormal solution of potassium permanganate. (Test solution should be made upto 100 ml)

#### Principle

The titration is based on the oxidation and the reduction reaction. The oxidising agent i.e Potassium permanganate oxidises the reducing agent ferrous sulphate and ferrous ammonium sulphate in acidic medium to ferric sulphate.

#### Procedure

#### Titration I:

#### Standardisation of Potassium permanganate

The burette is washed with water, rinsed with distilled water and then with the given Potassium permanganate solution. It is filled with same solution upto zero mark. The initial reading of the burette is noted. A 20ml pipette is washed with water, rinsed with distilled water and then with the given ferrous sulphate solution.20 ml of ferrous sulphate solution is pipetted out in to a clean conical flask. One test tube full of dilute sulphuric acid (20 ml) is added to it. It is titrated against Potassium permanganate taken in the burette. Potassium permanganate acts as the self indicator. The end point of the titration is the appearance of permanent pale pink colour. The titration is repeated to get the concordant value. From the titre value, the normality of Potassium permanganate solution is calculated.

#### Titration II:

#### Standardisation of Mohr's salt (ferrous ammonium sulphate)

The given Mohr's salt solution is made upto 100 ml in a 100 ml standard flask. The solution is thoroughly shaken to get a uniformly concentrated solution. A 20 ml pipette is washed with water, rinsed with distilled water and then with the given Mohr's salt solution .Using the rinsed pipette, exactly 20 ml of the made-up solution is transferred into a clean conical flask. To this solution one test tube full of dilute sulphuric acid (20 ml) is added. The solution is titrated against standardised

=.....N

potassium permanganate taken in the burette. The end point of the titration is the appearance of permanent pale pink colour. The titration is repeated to get the concordant value. From the titre value, the normality of given Mohr's salt solution and the amount of Mohr's salt in 100 ml of the given solution is calculated.

#### Result

1.Normality of Potassium permanganate

2.Normality of Mohr's salt (ferrous ammonium sulphate) =.....N

3.Amount of Mohr's salt present in 100 ml of given solution =.....g

## Titration-I : Potassium permanganate Vs Ferrous sulphate

S.No	Volume of Ferrous sulphate (ml)	Burette reading (ml)		Volume of Potassium	Indicator
		initial	final	(ml)	

#### Concordant value=

## Calculation:

Volume of Ferrous sulphate	$(V_1) = 20  \text{ml}$
Normality of Ferrous sulphate	$(N_1) =$
Volume of Potassium permanganate	$(V_2) =$
Normality of Potassium permanganate	$(N_2) = ?$
By the principle of volumetric analysis,	$V_1 N_1 = V_2 N_2$
: Normality of Potassium permanganate $(N_2)$ =	N

$$\therefore N_2 = \frac{V_1 N_1}{V_2} = \frac{X20}{V_2} = ----N$$

## Titration-II : Potassium permanganate Vs Mohr's salt

S.No	Volume of FAS (ml)	Burette reading (ml)		Volume of Potassium	Indicator
		initial	final	(ml)	

#### Concordant value=

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## **Calculation:**

Volume of Mohr's salt	(V <sub>1</sub> )	=20 ml

Normality of Mohr's salt  $(N_1) = ?$ 

Volume of Potassium permanganate  $(V_2)$  =

Normality of Potassium permanganate  $(N_2)$  =

By the principle of volumetric analysis,  $V_1N_1 = V_2N_2$ 

$$\therefore N_1 = \frac{V_2 N_2}{V_1} = \frac{X}{20} = - - - - N$$

 $\therefore \text{Normality of Mohr's salt} = \_$ Amount of Mohr's salt present }
in 100 ml of the given solution }

=----g

## SHORT PROCEDURE

Description	Titration I	Titration II
Burette solution Pipette solution Reagents added Indicator End point	Potassium permanganate Ferrous sulphate One test tube of dilute sulphuric acid self Appearance of pale permanent pink colour	Potassium permanganate Ferrous ammonium sulphate (mohr's salt) One test tube of dilute sulphuric acid self Appearance of pale permanent pink colour
Equivale	ent mass of Ferrous ammo	onium sulphate = 392

# 6. ESTIMATION OF FERROUS SULPHATE

#### EX.NO.....

#### Date.....

#### Aim

To estimate the amount of crystalline ferrous sulphate present in 500 ml of the given solution. You are provided with a standard solution of crystalline ferrous ammonium sulphate of normality .....N and an approximately decinormal solution of potassium permanganate.

#### Principle

The titration is based on the oxidation and the reduction reaction. The oxidising agent i.e Potassium permanganate oxidises both ferrous sulphate and ferrous ammonium sulphate in acidic medium to ferric sulphate.

## Procedure

#### Titration I:

#### Standardisation of Potassium permanganate

The burette is washed with water, rinsed with distilled water and then with the given Potassium permanganate solution. It is filled with same solution upto zero mark. The initial reading of the burette is noted. A 20ml pipette is washed with water, rinsed with distilled water and then with the given ferrous ammonium sulphate solution.20 ml of ferrous ammonium sulphate solution is pipetted out in to a clean conical flask.One test tube full of dilute sulphuric acid is added to it. It is titrated against Potassium permanganate taken in the burette. Potassium permanganate acts as the self indicator. The end point of the titration is the appearance of permanent pale pink colour. The titration is repeated to get the concordant value. From the titre value, the normality of Potassium permanganate solution is calculated.

## Titration II:

#### Standardisation of ferrous sulphate

The given ferrous sulphate solution is made upto 100 ml in a 100 ml standard flask. The solution is thoroughly shaken to get a uniformly concentrated solution. A 20 ml pipette is washed with water, rinsed with distilled water and then with the given ferrous sulphate solution. Using the rinsed pipette, exactly 20 ml of the made-up solution is transferred into a

clean conical flask. To this solution one test tube full of dilute sulphuric acid (20 ml) is added. The solution is titrated against standardised potassium permanganate taken in the burette. The end point of the titration is the appearance of permanent pale pink colour. The titration is repeated to get the concordant value. From the titre value, the normality of given ferrous sulphate solution and the amount of ferrous sulphate in 500 ml of the given solution is calculated.

## Result

1.Normality of Potassium permanganate

=.....N

2. Normality of ferrous sulphate

3. Amount of ferrous sulphate present in 500 ml of given solution=...g

Titration-I: Potassium permanganate Vs Mohr's salt (FAS)

S.No	Volume of FAS	Burette reading (ml)		Solume of Potassium	Indicator
	(ml)	initial	final	(ml)	

## Concordant value=

## Calculation:

Volume of FAS	$(V_1)$	=20 ml	
Normality of FAS	(N <sub>1</sub> )	=	
Volume of Potassium permanganate	$(V_2)$	=	
Normality of Potassium permanganate	$(N_2)$	=	?>
By the principle of volumetric analysis,	$V_1 N_1$	$= V_2 N_2$	

$$\therefore N_2 = \frac{V_1 N_1}{V_2} = \frac{X20}{V_1} = ----N$$

 $\therefore$  Normality of Potassium permanganate (N<sub>2</sub>) =

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S.No	Volume Ferrous sulphate (ml)	Burette reading (ml)		Solume of Potassium	Indicator
		initial	final	(ml)	

## Titration-II: Potassium permanganate Vs ferrous sulphate

## Concordant value=

## Calculation:

Volume of ferrous sulphate $(V_1)$	=	20 ml	
Normality of ferrous sulphate (N1)	=	?	
Volume of Potassium permanganate $(V_2)$	=		
Normality of Potassium permanganate $(N_2)$	=		
By the principle of volumetric analysis,	$V_1 N_1 = V_2 N_2$		
Normality of ferrous sulphate	=		Ν

: Normality of ferrous sulphate

$$:: N_1 = \frac{V_2 N_2}{V_1} = \frac{X}{20} = - - - - N$$

Amount of ferrous sulphate present in 500 ml of the given solution

= Equivalent mass x Normality of ferrous sulphate x 500/1000 = -----g

## SHORT PROCEDURE

Description	Titration I	Titration II
Burettesolution	Potassium permanganate	Potassium permanganate
Pipette solution	Ferrous ammonium sulphate	Ferrous sulphate
Reagents added Indicator End point	One test tube of dilute sulphuric acid self Appearance of pale permanent pink colour	One test tube of dilute sulphuric acid self Appearance of pale permanent pink colour
Equi	ivalent mass of Ferrous si	ulphate = 278

# 7. ESTIMATION OF TOTAL HARDNESSS OF WATER

#### Ex no.....

## Date.....

#### Aim:

To estimate the total hardness of the given sample of water by EDTA titration.

## **Principle:**

The total harness of water can be determined by titrating a known volume of hard water against EDTA solution using Erriochrome Black - T indicator. The estimation is based on the complexometric titration.

#### Procedure:

#### Titration-I

## Standarisation of EDTA solution

50 ml of the given calcium chloride solution is pipetted into a clean conical flask. Half a test tube of ammonia buffer solution is added into the conical flask. A pinch of Erriochrome Black - T indicator is added into the conical flask. The solution turns wine red in colour. It is tirated against EDTA solution taken in a clean, rinsed burette. The end point is the change in colour from wine red to steel blue. The tiration is repeated to get concordant values. From the titre values and the molarity of calcium chloride solution, the molarity of EDTA is calculated.

#### Tiration-II

#### Standarisation of hard water

50 ml of the given sample of hard water is pipetted into a clean conical flask. Half a test tube of ammonia buffer solution is added into the conical flask. A pinch of Erriochrome Black - T indicator is added into the conical flask. The solution turns wine red in colour. It is tirated against EDTA solution taken in a clean, rinsed burette. The end point is the change in colour from wine red to steel blue. The tiration is repeated to get concordant values. From the titre values, the hardness of the given sample of water is calculated in ppm.

#### **Result:**

The total harness of the given sample of water = ...... Ppm

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Fitration-I: Standard	calcium chloride	solution Vs EDTA

S.No	Volume of Standard calcium chloride	Burette reading (ml)		Volume of	Indicator
	sulphate (ml)	initial	final		

## Concordant value=

## Calculation

Volume of standard calcium chloride solution( $V_1$ )	= 50 (ml)
Molarity of standard calcium chloride solution( $M_1$ )	=0.01 M
Volume of EDTA solution $(V_2)$	= x (ml)
Molarity of EDTA solution (M2)	=

By the principle of volumetric analysis,  $V_1M_1 = V_2M_2$ 

$$\therefore M_2 = V_1 M_1 / V_2 = \frac{50 \times 0.01}{----}$$

 $\therefore$  The molarity of EDTA solution = .....M

## Standardised EDTA Vs Hard water sample

S.No	Volume of water sample (ml)	Burette reading (ml)		Volume of	Indicator
		initial	final		

#### Concordant value=

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## Calculation

Volume of hard water sample(
$$V_{hardwater}$$
) = 50 (ml)  
Volume of EDTA solution ( $V_{EDTA}$ ) = x (ml)  
Molarity of EDTA solution ( $M_{EDTA}$ ) =  
 $\therefore$  Total hardness =  $V_{EDTA} \times (M_{EDTA} / V_{hardwater}) \times 10^{6}$   
= ppm

## SHORT PROCEDURE

Description	Titration I	Titration II	
Burette	EDTA	EDTA	
Pipette solution	Calcium chloride solution	Hard water	
Reagents			
added	Buffer solution	Buffer solution	
Indicator	Eriochrome black-T	Eriochrome black-T	
End point	Change in colour from wine red to steel blue	Change in colour from wine red to steel blue	

# 8. DETERMINATION OF pH AND CALCULATION OF HYDROGEN ION CONCENTRATION

Ex. No.....

Date.....

#### Aim:

## To find out

- 1. The pH of given solutions in bottles A, B, C, D and E
- 2. To calculate hydrogen ion concentration of the solutions.

## Principle:

The pH of the solution can be directly measured using a pH meter. Acids give hydrogen ions in solution. The acidic nature of the solution depends on the hydrogen ion concentration which is expressed as gram ions per litre. The pH of the solution varies with concentration of hydrogen ions.

$$PH = -\log_{10}[H^{+}]$$

## Procedure:

Exactly 50 ml of the given five sample solutions are taken in five 150 ml beakers and labeled as A,B,C,D and E. The pH meter is standardized using a known buffer solution. The electrodes are then washed with distilled water and then immersed in the solution taken in the beaker.

The pH readings are noted. The pH of all the other solutions are to be determined similarly. The electrodes are washed well with distilled water before the electrodes are immersed in next solution. The amount of hydrogen ions present in the solutions are then calculated from the pH.

## **Result:**

The amount of pH and hydrogen ion concentration of the given five sample solutions are

## pH and [H<sup>⁺</sup>]CONCENTRATION

(1) Sample A ...... g ions / lit
(2) Sample B ..... g ions / lit
(3) Sample C ..... g ions / lit
(4) Sample D ..... g ions / lit
(5) Sample E ..... g ions / lit

## DETERMINATION OF pH AND CALCULATION OF HYDROGEN ION CONCENTRATION

S.No	Sample solution	рН	Hydrogen ion concentration g ions per litre
1	А		
2	В		
3	С		
4	D		
5	E		

Calculations

Sample A

#### Sample B

Sample C

Sample D

Sample E

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## MODEL QUESTION PAPER

## MODEL: 1

1. Estimate the mass of Sulphuric acid Present in 500 ml of the given solution. You are supplied with a standard solution of oxalic acid of strength 0.098N and an approximately decinormal solution of Sodium hydroxide.

#### MODEL: 2

2. Calculate the total hardness of the given sample of water. You are given a standard Hard water Solution of 0.01M and an approximately 0.01M EDTA solution.

#### MODEL: 3

3. Calculate pH of given five samples, using pH meter and Calculate the  $H^+$  ion Concentration of all the samples. (Any two Students only in a batch).

## List of Apparatus to be supplied for each student for Board Exam

1.	Burette 50ml	- 1
2.	Pipette 20ml (with safety bulb)	- 1
3.	Conical Flask 250ml	- 1
4.	Funnel	- 1
5.	Porcelain Tile 6x6"	- 1
6.	Burette stand	- 1
7.	Standard flask 100 ml	- 1
8.	Beakers 250 ml	- 1
9.	Wash Bottle	- 1

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