



## **Unit1: WATER**

▪ **Introduction:**

Earth's 71% surface area is acquired by water and remaining 29% by land out of that 71% ocean contains 97% of water, 2% is in ice form and only 1% is good for use. Ocean water can't be used for drinking, industrial or agricultural use as it is saline. Hence we are totally depends on rain water. The quality of water is important as concern to human health. Water play important role in various life process in human body.

**Sources of water:**

The sources of water are classified as,

- A) Rain Water
- B) Surface water- Lake water, River water, Sea water, Lake water
- C) Underground water

Surface water is water which falls to the ground as rain or hail. This water is collected from special area called catchment. The catchment feeds water into a holding area via rivers, streams and creeks. The water is then stored in a natural or artificial (manmade) barrier called a dam or reservoir. Dams are usually placed at the lower end of a valley.

▪ **Impurities in natural water:**

Contamination of natural water is mostly due to- Solid or Suspended Particles, Oils, Inorganic or organic Salts, Biological Impurities and Gases like  $\text{Cl}_2$ ,  $\text{CO}_2$ ,  $\text{SO}_2$ ,  $\text{O}_2$  etc.

A) Impurities present in rain water, surface water and underground water.

- i) Rain water is the purest form of naturally occurring water. But when it falls on earth's surface and floats, it gets contaminated due to presence of several types of organic, inorganic matter and gases gets mixed in it.
- ii) Surface water can be sub classified as river water, sea water & lake water. River water contains dissolved minerals like bicarbonates of calcium, magnesium and iron. Also small particles of rock, sand and organic matter derived from decomposition of plants and animals present in river water. Sea water is the most impure form water which contains organic and inorganic matter, added from river, domestic areas, industrial wastes, etc. lake water is contains organic matter in quite high quantity.
- iii) Underground water contains minerals, oil globules, sand, soil decayed organic matter as contaminants.

B) Solid or Suspended Particles:-

- i) Solid particles (size greater than  $10^{-4}$  cm) lighter than water are present in natural water as suspended impurities which floats on the surface and suspended inside the water.



e.g. pieces of woods, leaves, branches of trees, pieces of other organic matters, mud, algae, industrial wastes, oils etc.

- ii) Due to this colour, odour, taste of water gets changed as well as it causes turbidity.
- iii) Some of these impurities may get settle down due to effect of gravitational force and remaining can be removed by sedimentation or by filtration.
- iv) If the size of solid particles lies between  $10^{-5}$  to  $10^{-7}$  cm then they are called as **colloidal impurities**. It is mainly due to clay particles, soil, silica, hydroxides of iron and aluminium, organic particles etc.
- v) They cause turbidity of water and cannot settle down with gravitational force effect. They can be removed by coagulation process.

C) Dissolved Impurities:-

- i) **Dissolved impurities** present in water are mostly due to inorganic substances, biological substances and various gases dissolved in natural water. e.g. Calcium and magnesium salts causes alkalinity, hardness. Metals and their oxides changes colour, poisoning, taste, hardness. Gases like  $\text{Cl}_2$ ,  $\text{CO}_2$ ,  $\text{SO}_2$ ,  $\text{H}_2\text{S}$ ,  $\text{O}_2$  etc. responsible for acidity, odour, Corrosion of metals.
- ii) These dissolved impurities in water can be removed by chemical treatments like ion exchange method, reverse osmosis, lime soda process etc.

D) Biological Impurities:-

- i) **Biological impurities** are suspended and dissolved. e.g. bacteria, disease producing microorganisms, dead animals, vegetables, organic wastes etc.
- ii) These type of impurities cause objectionable odour, taste, acidity, diseases etc. They can be removed by filtration, sterilization etc.

▪ **Water Testing Methods:**

A) Total dissolved solid (TDS)

1. Solids present in water are suspended solid, fixed & volatile solids, settleable solids.
2. Because of presence of these type of solid water becomes dirty, affect colour & odour of water thus such contaminated water is unacceptable
3. Determination of total solids (dried at  $103-105^\circ\text{C}$ ):
  - a. A preweighted nickel crucible is taken in which a aliquot (corresponding to about 0.1 gram of dissolved matter) of well mixed water is transferred.
  - b. Then nickel crucible is evaporated to dryness on a water bath.
  - c. Then crucible is dried in an electric oven at  $103-105^\circ\text{C}$  for 1 hrs, cool in desiccators & weighed.
  - d. Repeated drying, cooling & weighing done until constant weight is obtained (i.e. weight loss less than 4% of previous wt.)
  - e. Let,

$$\text{Volume of sample taken} = V \text{ ml}$$



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Department of First Year Engineering

Subject: Engineering Chemistry

Weight of empty crucible =  $W_1$  gm

Weight of crucible + residue =  $W_2$  gm

(Dried at 103-105°C)

So weight of solid in sample =  $(W_2 - W_1)$  gm

Therefore, Total dissolved solid =  $\frac{W_2 - W_1}{V}$  gm/lit

## B) Acidity

1. (defn) It is defined as capacity to combine with hydroxyl ions to form a molecule of water. It is the measure of acids present in water.
  2. Neutral water becomes acidic due to the addition of a) acidic salts b) acidic gases & c) acidic industrial water
  3. If  $\text{CO}_2$  is present then it forms carbonic acid with water & water becomes acidic
    - a.  $\text{CO}_2 + \text{H}_2\text{O} \rightarrow \text{H}_2\text{CO}_3$
- Similarly if  $\text{H}_2\text{S}$  is present it forms sulphuric acid. Some organic materials on decomposition form the organic acid. Also due to addition of minerals acids like  $\text{Fe(OH)}_3$ ,  $\text{Al(OH)}_3$  water becomes acidic.
4. Acidic water is unsuitable for domestic and industrial application. It increases corrosion of metal. Therefore it is necessary to find out acidity of water. If water is acidic it can be neutralized by adding alkali.
  5. The acidity of water is determined by acid base titration.
  6. Water is titrated with standard alkali solution in presence of indicator phenolphthalein ( $\text{pH} = 8.3$ ) & methyl orange ( $\text{pH} = 4.5$ )
  7. Phenolphthalein measures total acidity of water while methyl orange determines mineral acidity of water.

### 8. Procedure:

#### a) Methyl orange acidity:

Rinse and fill the burette with given standard N/50 NaOH Solution. Pipette out 25 ml of water sample in a clean titration flask. Add 1 to 2 drops of methyl orange indicator. The solution becomes orange in colour. Titrate this against N/50 NaOH solution from the burette with drop by drop and constant shaking of flask till the orange colour changes to yellow. Note this burette reading as methyl orange end point and repeat the same procedure for three times to get constant burette reading (A).

#### b) Phenolphthalein acidity:

Repeat the same procedure by changing indicator i.e. add 3-4 drops of phenolphthalein indicator, titrate it against N/50 NaOH until pink colour appears. Note this burette reading as phenolphthalein end point and repeat procedure for three times to get constant burette reading (B).

### 9. Calculation:

Acidity of sample determined by methyl orange Indicator.

$$\text{mg/lit as CaCO}_3 = \frac{A \times \text{normality of NaOH} \times 1000 \times 50}{\text{Volume of Sample}}$$



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= \_\_\_\_\_ ppm

Acidity of sample determined by phenolphthalein indicator.

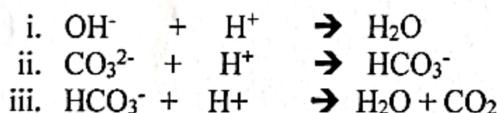
$$\text{mg/lit as CaCO}_3 = \frac{B \times \text{normality of NaOH} \times 1000 \times 50}{\text{Volume of Sample}}$$
$$= \text{_____ ppm}$$

Total acidity of water sample at pH 8.3

$$\text{mg/lit as CaCO}_3 = \frac{(A+B) \times \text{normality of NaOH} \times 1000 \times 50}{\text{Volume of Sample}}$$
$$= \text{_____ ppm}$$

## C) Alkalinity

- 1 Total content of those substances that causes an increased concentration of  $\text{OH}^-$  ions upon dissociation or due to hydrolysis is called alkalinity of water.
- 2 This alkalinity is due to presence of following constituents  $\text{OH}^-$  only,  $\text{CO}_3^{2-}$  only,  $\text{HCO}_3^-$  only,  $\text{OH}^-$  &  $\text{CO}_3^{2-}$ ,  $\text{CO}_3^{2-}$  &  $\text{HCO}_3^-$
- 3 There is no possibility of hydroxide & bicarbonates existing together as,
- 4 Water becomes alkaline due to addition of alkaline salt from soil & industries, alkaline industrial waste water & alkaline gases like ammonia.
- 5 Alkalinity of water can be determined by using acid base titration in which the water sample is titrated against standard acid in presence of phenolphthalein & methyl orange indicator.
- 6 Determination of alkalinity is based on the following chemical reaction.



- 7 The titration of the water sample against a standard acid up to phenolphthalein end point determines hydroxides and half of the normal carbonate present
- 8 On the other hand titration of water against standard acid to methyl orange end point determines the remaining half of the normal carbonate and all the bicarbonates. The total amount of acid represents the total alkalinity due to hydroxide, bicarbonate & carbonate ions.
- 9 Alkaline water is unsuitable for use in industries. It increases the rate of corrosion of metals. The alkalinity of water can be adjusted with addition of acids.

## 10. Procedure:

- a) Rinse and fill the burette with given standard N/50  $\text{H}_2\text{SO}_4$  solution.
- b) Pipette out 25 ml of water sample in a clean titration flask. To that solution add a 2 to 3 drop of phenolphthalein solution becomes pink in colour.



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- c) Titrate this solution against N/50  $\text{H}_2\text{SO}_4$  solution from the burette with drop by drop and constant shaking of flask till the pink colour just vanishes.
- d) Note this burette reading as phenolphthalein end point  $V_1$  ml
- e) Then to the same solution in titration flask add 2 to 3 drops of methyl orange indicator, solution becomes yellow in colour.
- f) Continue the titration with addition of N/50  $\text{H}_2\text{SO}_4$  solution from the burette with drop by drop and constant stirring of flask till the yellow colour changes to orange.
- g) Note this extra volume of acid as  $V_2$  ml so that methyl orange end point becomes  $V_1 + V_2$  ml.

## 11. OBSERVATIONS:

1. Volume of acid used to phenolphthalein end point = .....  $V_1$  ml
2. Volume of acid used to methyl orange end point = .....  $V_2$  ml

## 12. Calculation:

### 1. Phenolphthalein Alkalinity-

$$P = \frac{V_1 \times \text{Normality of } \text{H}_2\text{SO}_4 \times 1000 \times 50}{\text{Volume of Water Sample}}$$

$$= \frac{1 \times (V_1) \times 1000 \times 50}{50 \times 25}$$

$$= 40 \times \dots \dots \dots (V_1) \text{ ppm}$$

$$= \dots \dots \dots \text{ ppm}$$

### 2. Methyl orange Alkalinity-

$$M = \frac{(V_1 + V_2) \times \text{Normality of } \text{H}_2\text{SO}_4 \times 1000 \times 50}{\text{Volume of Water Sample}}$$

$$= \frac{1 \times (V_1 + V_2) \times 1000 \times 50}{50 \times 25}$$

$$= 40 \times \dots \dots \dots (V_1 + V_2) \text{ ppm}$$

$$= \dots \dots \dots \text{ ppm}$$

## Calculation of Concentrations of $\text{OH}^-$ , $\text{CO}_3^{2-}$ and $\text{HCO}_3^-$ ions:-

Alkalinity	$\text{OH}^-$ ppm	$\text{CO}_3^{2-}$	$\text{HCO}_3^-$
$P=0$	0	0	M
$P=1/2M$	0	$2P$	0
$P < 1/2 M$	0	$2P$	$M-2P$
$P > 1/2 M$	$2P-M$	$2(M-P)$	0
$P=M$	M	0	0

For the given sample of water ..... (Condition), therefore the given water contains ..... and ..... ions only. Thus,

1. Alkalinity due to  $\text{OH}^-$  ions = = ppm,
2. Alkalinity due to  $\text{CO}_3^{2-}$  ions = = ppm,
3. Alkalinity due to  $\text{HCO}_3^-$  ions = = ppm.

Result of titration	Hydroxide( $\text{OH}^-$ ) $V_1$ in ml	Carbonate ( $\text{CO}_3^{2-}$ ) $V_2$ in ml	Bicarbonate ( $\text{HCO}_3^-$ ) $V_3$ in ml
$P=0$	Nil	Nil	M
$P=1/2M$	Nil	$2P$	Nil



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P>1/2 M	2P-M	2(M-P)	Nil
P<1/2 M	Nil	2P	(M-2P)
P=M	M	Nil	Nil

- i) Check the condition satisfied by P & M  
ii) Then,

$$\text{Alkalinity due to hydroxide } (\text{OH}^-) = \frac{V_1 \times 100}{V} = \text{mg/lit}$$

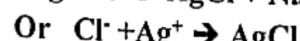
$$\text{Alkalinity due to carbonate } (\text{CO}_3^{2-}) = \frac{V_2 \times 100}{V} = \text{mg/lit}$$

$$\text{Alkalinity due to bicarbonate } (\text{HCO}_3^-) = \frac{V_3 \times 100}{V} = \text{mg/lit}$$

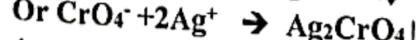
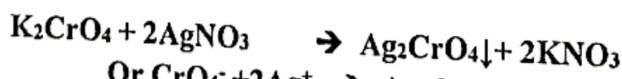
Total alkalinity is sum of all above values.

E) Chloride content

- 1) The chloride present in the water is the form of  $\text{CaCl}_2$ ,  $\text{MgCl}_2$ ,  $\text{NaCl}$  & free chlorine.
- 2) Drinking water is disinfected with chlorine to remove the bacteria, fungi and other microorganisms.
- 3) If chlorine is present beyond the certain limit, it becomes unfit for drinking purposes & produces bad odour. Also chloride content in water increases the rate of corrosion of metals. Therefore it is necessary to estimate the chloride content of water.
- 4) The chloride content in water can be determined by using precipitation titration. Precipitation titration is titration in which one of the product obtained is in the form of precipitate.
- 5) To determine chloride content water is titrated against standard solution of silver nitrate in presence of potassium chromate or sodium chromate as an indicator.
- 6)  $\text{AgNO}_3$  precipitate the chloride ions as silver chloride



- 7) When chloride ions get removed completely the excess silver nitrate react with potassium chromate & forms brick red precipitate of silver chromate, which indicates end point of reaction.



- 8) This titration is to be carried out at  $\text{pH}=7$  because the precipitation of chloride ions with silver nitrate is possible only in neutral solution. If the water sample is acidic, it can be neutralized by adding chloride free sodium carbonate.
- 9) Procedure:



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Wash the apparatus cleanly with distilled water. Fill the burette with 0.01N  $\text{AgNO}_3$ . Pipette out 10ml of water sample in conical flask then add 2 to 3 drops of potassium chromate as in indicator. Then titrate the water sample with 0.01 N  $\text{AgNO}_3$  till colour changes from yellow to reddish brown. Take three readings and find the mean burette reading (V ml).

### 10) Calculation:

The Chloride content in given water sample 1 & 2 respectively,

$$\begin{aligned}
 &= \frac{V \text{ ml} \times \text{Normality of } \text{AgNO}_3 \times 1000 \times 35.5}{\text{Volume of Water Sample}} \\
 &= \frac{V \text{ ml} \times 0.01 \times 1000 \times 35.5}{10} \\
 &= \dots \text{ ppm.}
 \end{aligned}
 \quad
 \begin{aligned}
 &= \frac{V \text{ ml} \times \text{Normality of } \text{AgNO}_3 \times 1000 \times 35.5}{\text{Volume of Water Sample}}
 \end{aligned}$$

### F) Dissolved Oxygen

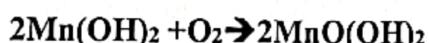
1. Oxygen is poorly soluble in water If sufficient D.O. is not present in polluted water anaerobic occur So D.O. test is vital as a means of controlling the rate of aeration which is also important for aquatic life.

#### 2. Theory :

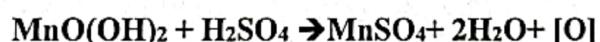
a. In dissolved oxygen test manganese oxide is formed by the reaction  $\text{NaOH}$  &  $\text{MnSO}_4$



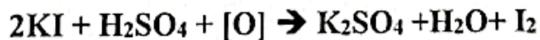
b. The D.O. present in water oxidizes  $\text{Mn}^{2+}$  to its higher valency.



c. This manganese oxide on reaction with acid reverts back to divalent state & gives molecular oxygen.



d. This molecular oxygen then takes part in reaction of  $\text{KI}$  with acid which liberate equivalent amount of water & iodine.



e. This iodine is treated against standard sodium thiosulphate using starch indicator.



3 Then,

- Collect 300 ml of water sample in BOD bottle avoiding contact of air.
- Add 2 ml of  $\text{MnSO}_4$  solution followed by 2 ml of alkaline iodide azide solution in such a way that the tip of pipette should deep below the liquid surface.
- Stopper the bottle immediately & mix well by inverting the bottle 3 to 4 times this causes precipitation Allow to stand for two minute.



- d. Remove the stopper & add 1 ml conc.  $H_2SO_4$  insert stopper & mix well till precipitate goes into solution Allow to stand for 5 min.
- e. Take 102 ml of this solution into a conical flask & titrate it against N/50  $Na_2S_2O_3$  solution till pale yellow coloration. Add few drops of starch indicator light blue blackish colour appear.
- f. Continue titration till blue colour disappears (V ml).

$$D.O. = (V/40)/(100 \times 8 \times 1000) \text{ mg/L or ppm}$$

▪ **Hardness of water:**

1. Hardness of water is "the soap consuming capacity of water sample".

or

hard water is defined as, " water which prevent lathering soap".

2. The hard water generally contains certain salt of calcium magnesium & other heavy metals dissolved in it.
3. These ions of salts react with sodium salts of long chain fatty acids present in soap lather is not formed but it forms insoluble white scum's or precipitates of calcium & magnesium which do not possess any detergent value. Other Metal  $Fe^{2+}$ ,  $Mg^{2+}$ ,  $Al^{3+}$  also react.
4. There are two types of hardness

A) **Temporary or carbonate or alkaline hardness.**

B) **Permanent or non-carbonate or non-alkaline hardness**

**A) Temporary or carbonate or alkaline hardness**

- i) Temporary hardness is due to carbonates, bicarbonates & hydroxide of calcium, magnesium & other metal.
- ii) Temporary hardness is mostly removed by simple physical process like by more boiling of water where bicarbonate decomposed producing insoluble carbonate or hydroxide.

**B) Permanent or non-carbonate or non-alkaline hardness**

- i) Permanent hardness is due to presence of dissolved chlorides, sulphates & nitrate of calcium, magnesium, iron & other heavy metals.
- ii) This can't be removed by applying simple procedure like boiling.
- iii) It requires chemical treatment such as external treatment or internal softening.
- iv) The external treatment involve softening method such lime soda method, zeolite permutite method, ion exchange method etc.
- v) Internal treatment involves by and large conditioning treatment. For this chemicals commonly used are EDTA, Lignin, Sodium hydrogen phosphates etc.



▪ Disadvantages of hard water: -

A) Domestic purpose:

- 1) **Washing:** It causes wastage of soap & forms sticky precipitate (Ca & Mg spots) which sticks to fabric /clothes giving spots.
- 2) **Bathing:** It causes wastage of soap, production of sticky scum on bucket / bath-tub & body. Thus cleaning quality of soap reduced & skin becomes dry & dark colors.
- 3) **Cooking:** Due to hardness of water, boiling point elevates which result into wastage of fuel & of more time for cooking.
- 4) **Drinking:** It causes harmful effects on digestive & urinary track systems.

B) Industrial purposes:

- 1) **Textile Industrial:** Hard water causes wastage of soap with formation of Ca & Mg soaps. These fabric when dyed latter, do not produce exact shade of the colour.
- 2) **Sugar Industry:** Hard water used sugar refining causes difficulty in crystallization of sugar. So sugar produced may undergo decomposition during storage.
- 3) **Paper Industry:** Ca & Mg salts react with chemicals which smooth & glossy finish to paper. Also iron salts may even affect the color of paper being produced.
- 4) **Laundry:** If hard water used for laundry, wastage of soap occurs.
- 5) **Concrete Making:** Hard water containing chlorides & sulphates affects hydration of cement & strength of concrete.
- 6) **Pharmaceutical Industry:** If hard water used in making drugs syrups, injection etc. then it may produce certain undesirable products.

▪ Calculations of total hardness (Numerical):

- 1) The water sample on analysis was found to contain the following impurities in mg/lit.  $\text{Ca}(\text{HCO}_3)_2$ -9.72 ppm,  $\text{Mg}(\text{HCO}_3)_2$ -20.44 ppm,  $\text{CaSO}_4$ - 16.32 ppm,  $\text{MgSO}_4$ -16.80 ppm,  $\text{CaCl}_2$ -17.76 ppm Calculate temporary, permanent and total hardness of water sample in degree Clark & comment on drinkability.

Sol<sup>n</sup>:  $\text{CaCO}_3$  equivalents in mg/lit are as follows,



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Name of Salt	Quantity in ppm	Multiplication Factor	CaCO <sub>3</sub> equivalents in ppm	Type of Hardness
Ca(HCO <sub>3</sub> ) <sub>2</sub>	9.72	9.72 x 100/162	6.0	Temporary
Mg(HCO <sub>3</sub> ) <sub>2</sub>	20.44	20.44 x 100/146	14.0	Temporary
CaSO <sub>4</sub>	16.32	16.32 x 100/136	12.0	Permanent
MgSO <sub>4</sub>	16.80	16.80 x 100/120	14.0	Permanent
CaCl <sub>2</sub>	17.76	17.76 x 100/111	16.0	Permanent

Now, Temporary hardness is due to,

$$\text{Ca}(\text{HCO}_3)_2 + \text{Mg}(\text{HCO}_3)_2 = 6.0 + 14.0 = 20.0 \text{ ppm} = 20.0/14.3 = 1.39 \text{ }^\circ\text{C}$$

Then Permanent hardness is due to,

$$\text{CaSO}_4 + \text{MgSO}_4 + \text{CaCl}_2 = 12.0 + 14.0 + 16.0 = 42.0 \text{ ppm} = 42.0/14.3 = 2.93 \text{ }^\circ\text{C}$$

Therefore, Total Hardness = Temporary Hardness + Permanent Hardness

$$= 20.0 + 42.0$$

$$= 62.0 \text{ ppm}$$

$$= 62.0 \times 0.07$$

$$= 4.32 \text{ }^\circ\text{C}$$

Answer:- 1) Temporary hardness = 1.39  $^\circ\text{C}$

2) Permanent hardness = 2.93  $^\circ\text{C}$

3) Total Hardness = 4.32  $^\circ\text{C}$

4) The given water sample can be suitable for drinking.

2. The water sample on analysis was found to contain the following salts in ppm.

Ca(HCO<sub>3</sub>)<sub>2</sub>-15.0 ppm, MgSO<sub>4</sub>- 16.32 ppm, MgCl<sub>2</sub>-14.8 ppm, CaCl<sub>2</sub>-19.0 ppm. Calculate temporary, permanent and total hardness of water sample in degree Clark & comment on drinkability.

Sol<sup>n</sup>: CaCO<sub>3</sub> equivalents in mg/lit are as follows,

Name of Salt	Quantity in ppm	Multiplication Factor	CaCO <sub>3</sub> equivalents in ppm	Type of Hardness
Ca(HCO <sub>3</sub> ) <sub>2</sub>	15.0	15.0 x 100/162	9.25	Temporary
MgSO <sub>4</sub>	16.32	16.32 x 100/120	13.6	Permanent
MgCl <sub>2</sub>	14.8	14.8 x 100/95	15.57	Permanent
CaCl <sub>2</sub>	19.0	19.0 x 100/111	17.11	Permanent
Ca(HCO <sub>3</sub> ) <sub>2</sub>	15.0	15.0 x 100/162	9.25	Temporary

Now, Temporary hardness is due to, Ca(HCO<sub>3</sub>)<sub>2</sub> = 9.25 ppm = 9.25 x 0.07 = 0.64 °Cl

Then Permanent hardness is due to,

$$\text{MgSO}_4 + \text{MgCl}_2 + \text{CaCl}_2 = 13.6 + 15.57 + 17.11$$

$$= 46.28 \text{ ppm}$$

$$= 46.28 \times 0.07 = 3.23 \text{ }^\circ\text{Cl}$$

Therefore, Total Hardness = Temporary Hardness + Permanent Hardness



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$$= 9.25 + 46.28$$

$$= 55.53 \text{ ppm} = 55.53 \times 0.07 = 3.88 \text{ °Cl}$$

Answer:- 1) Temporary hardness = 0.64 °Cl

2) Permanent hardness = 3.23 °Cl

3) Total Hardness = 3.88 °Cl

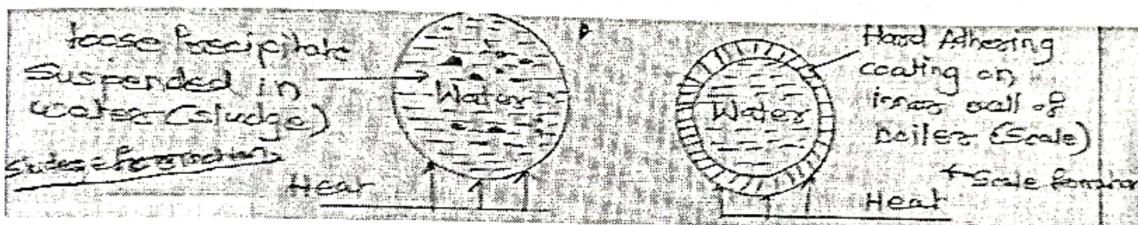
4) The given water sample may be suitable for drinking purpose.

## Effects of hard water-

Boilers employed for steam generation in number of industries & in power houses. On the basis of pressure ranges boilers are classified as low pressure, medium pressure and high pressure boilers. When hard water is directly fed into boilers problems occur as follows.

**I] Sludge & Scale formation:** Water is continuously evaporated to form steam in boilers. This evaporation increases concentration of dissolved salts & they are thrown out as precipitates.

- 1) These precipitate are soft, slimy & floating, these are known as **sludge**.
- 2) While if the precipitate is hard & forms coating on inner walls of boilers tubes, it is called as **scale**.



### A) Sludge:

- 1) Sludge is soft, loose & slimy precipitate formed within the boilers.
- 2) Sludge's are formed by substances which have greater solubility's in water than in cold water, e.g.  $\text{MgCO}_3$ ,  $\text{MgCl}_2$ ,  $\text{CaCl}_2$ ,  $\text{MgSO}_4$  etc.
- 3) Sludge's are formed in comparatively colder areas of boilers & are collected in areas where flow rate is slow or at bends in the pipes. This may lead to choking of pipes.

### Disadvantage:

- i) Sludge's are poor conductors of heat.
- ii) If sludges are formed along with scales, then former gets entrapped in latter & both get deposited as scales.
- iii) Excessive sludge's if formed in boilers, choke up pipe connection, plug opening & disturb the working of boilers.

### Prevention:

- i) Using the soft water in boilers.
- ii) Frequent blow down operation i.e. drawing off a portion of the concentrated water (containing large amount of dissolved salts) & replacing it by fresh water.

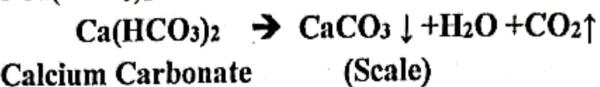


### B) Scales :- (Formation of scales)

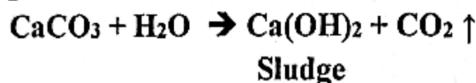
**B) Scales :** (Formation of scale) -  
Scales are hard deposits which stick very firmly to the inner surface of boilers. As scales are hard & adherent it is difficult to remove them even with help of hammer and chisel.

### **Cause of scale Formation:-**

i) Decomposition of  $\text{Ca}(\text{HCO}_3)_2$



The  $\text{CaCO}_3$  formed is soft. In low pressure boilers, it is the main cause of scale formation, however  $\text{CaCO}_3$  reacts to give  $\text{Ca}(\text{OH})_2$  as sludge and  $\text{CO}_2$  which gets evaporated.

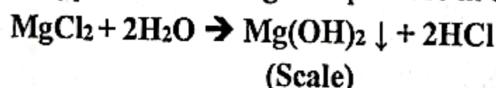


## ii) Deposition of $\text{CaSO}_4$ :-

In high pressure boilers it is main cause of scale formation. It is adherent difficult to remove. When hard water containing  $\text{CaSO}_4$  is heated  $\text{CaSO}_4$  precipitated as hard scale. The reason is solubility of  $\text{CaSO}_4$  high in cold water and it is practically insoluble in hot water.

## ii) Hydrolysis of Magnesium Salts:-

Magnesium salts form a soft type of scale at high temperature in boilers.



iii) Presence of Silica ( $\text{SiO}_3$ )-

Silica reacts with calcium and magnesium metals to form  $\text{CaSiO}_3$  & / or  $\text{MgSiO}_3$  which gets deposited on inner side of the boiler surface. They are highly sticky & forms hard layer on inner walls so difficult to remove.

### **Disadvantages:-**

- i) Wastage of Fuels:- When scale get adhered to inner walls of boiler tube the rate of heat transfer from wall of boiler to water is decreased greatly. To get steady supply of heat to water, overheating is done and this increase fuel consumption.
  - ii) Decrease in Efficiency: Scales may get deposited in the valves and condensers of boilers & can choke them partially, results in decrease in efficiency.
  - iii) Lowering of boiler safety: due to scale formation on inner walls of boiler tubes overheating of tube is done which makes the boiler material softer & weaker. This causes distortion of boiler tubes & makes the boiler unsafe to bear the pressure of steam.



- iv) Danger of Explosion:- The boiler is provided with high amount of heat due to scales the same amount of heat is not transmitted to water inside. Metal parts of boiler as well as layer of scales undergo expansion. Later stage comes when thick scales crack due to uneven expansion and hot water comes in contact with inner walls of boiler tube. As a result sudden production of large amount of steam takes place which leads to sudden increase in pressure which causes explosion of boiler.

### Removal of Scales:-

- If scales are loosely adhering, then can be removed by scraping with a piece of wood or wire brush.
- If scales are brittle by giving thermal shocks (sudden heating & cooling) they can be removed.
- If scales are hard & adherent they can be removed by dissolving them in suitable chemicals.

### Difference in between sludge and scales:-

Sr. No.	Sludges	Scales
1	Sludges are soft, loose and slimy precipitate.	Scales are hard deposits
2	They are non-adherent deposits and can be removed easily.	They stick very firmly to the inner surface of boiler and are very difficult to remove.
3	Formed by substances like $\text{CaCl}_2$ , $\text{MgCl}_2$ , $\text{MgSO}_4$ , $\text{MgCO}_3$	Formed by substances like $\text{CaSO}_4$ , $\text{Mg(OH)}_2$ etc.
4	Formed at comparatively colder portions of the boiler.	Formed at generally at heated portions of boiler.
5	They decrease the efficiency of boiler but are less dangerous.	Decrease the efficiency of boiler and chances of explosion are also there.
6	Can be removed by blow-down operation.	Cannot be removed by blow down operation.

#### • Treatments of hard water:

For industrial purposes efficiently pure, free from all hardness producing salt especially  $\text{Ca}$  &  $\text{Mg}$  salts water is used. The purpose of removing or reducing concentrate hardness causing salt from water is called softening of water.

Number of methods can be used for conversion of hard water to soft water.

I] Ion exchange process.

II] Reverse osmosis process



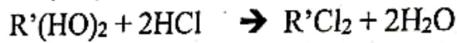
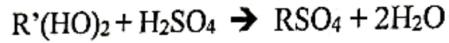
**I] Ion exchange process:**

- 1) In ion exchange process, ion exchange resins are used which are insoluble, cross linked, long chain, high molecular weight organic polymers.
  - 2) In this a reversible exchange of ions occurs between the stationary ion exchange phase & external liquid mobile phase.
  - 3) Functional group decides nature of resins exchanger i.e. cationic or anionic.
  - 4) On the basis of acidity or basicity they are classified below:-
- A) **Cation exchange resin(RH<sub>2</sub>/(RH<sup>+</sup>):-**
- Resins containing acidic functional groups (-COOH, -SO<sub>3</sub>H) exchange their H<sup>+</sup> ions with other cations, which come in their contact are known as cation exchange resin.
- Their exchange reactions with cation [Ca<sup>2+</sup> & Mg<sup>2+</sup>]
- $$RH_2 + Ca^{2+} \rightarrow RCa + 2H^+$$
- $$RH_2 + Mg^{2+} \rightarrow RMg + H^+$$

B) **Anion exchange resin [R'(OH)]:-**

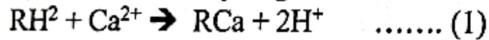
The Resins containing basic functional groups (e.g. -NH<sub>2</sub>, =NH etc.) exchange their anion other anions which come in their contact are called as anion exchange resins.

Their exchange reactions with cation [SO<sub>4</sub><sup>2-</sup>, CO<sub>3</sub><sup>2-</sup> etc] are



5) **Principle:**

- i) When the hard water is passed through cation exchange which remove all cation like Ca<sup>2+</sup>, Mg<sup>2+</sup>, Na<sup>+</sup> & release hydrogen ion



- ii) The sulphates, chlorides, carbonates into acids H<sub>2</sub>SO<sub>4</sub>, HCl,

- iii) After this acidic water is passed through an anion exchange bed with which remove all anion like SO<sub>4</sub><sup>2-</sup>, Cl<sup>-</sup> & equivalent amount of OH<sup>-</sup> ions are released



- iv) H<sup>+</sup> & OH<sup>-</sup> ions are released from cation exchange & anion exchange bed respectively get combine to produce water molecule.



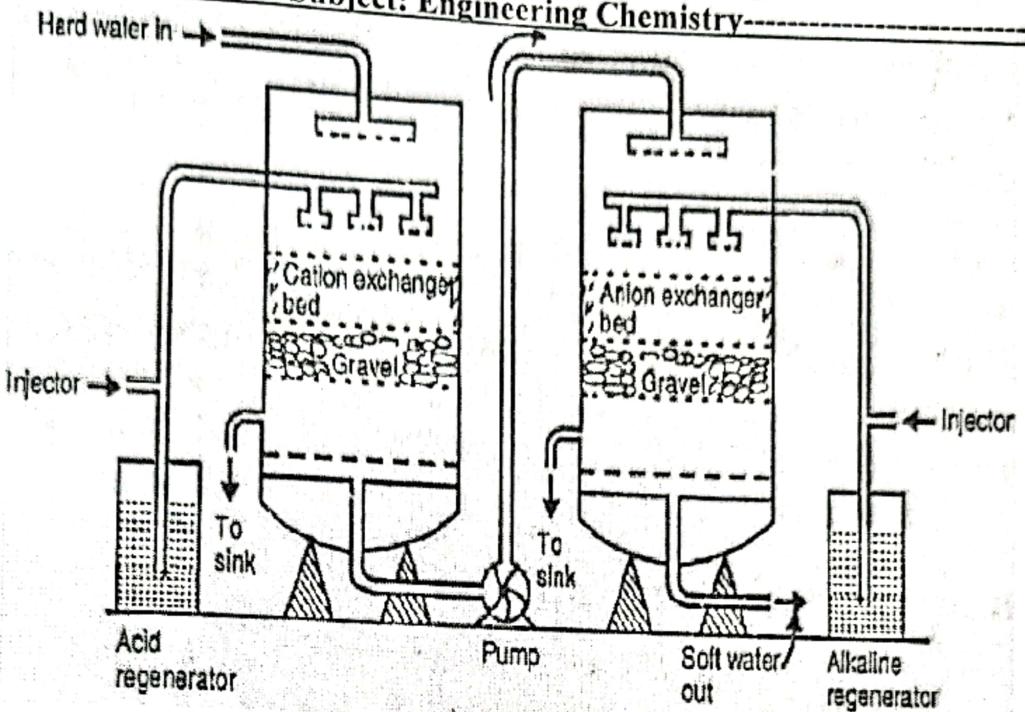
The resulting ion free water is deionized water or demineralised water



# D. Y. Patil College of Engineering and Technology

Approved by AICTE, New Delhi, Recognized by Govt. of Maharashtra & Affiliated to Shivaji University, Kolhapur

(An Autonomous Institute)  
Department of First Year Engineering  
Subject: Engineering Chemistry



- 6) The capability of ion exchange of ion exchange resins to exchange ion from hard water is based on ion exchange capacity. So regeneration of exhausted resin is done

#### Advantage

- Highly acidic or alkaline water can be softened.
- It produce water of low hardness (up to 2 ppm)

#### Disadvantage

- Equipment is costly.
- If the water is turbid then output is reduced.

## II] Reverse Osmosis:-

- The process of removing common salt (sodium chloride) from the water is known as desalination.
- The water containing dissolved salts with a peculiar salty (or brackish) taste is brackish water. Sea water containing on an average about 3.5 % salts, comes under this category. Brackish water is totally unfit for drinking purpose.
- Commonly used methods for the desalination of brackish water are Reverse Osmosis & electrodialysis. (out of which only Reverse osmosis is given below)
- Principle of Reverse Osmosis:-**

  - Two solutions of unequal concentrations are separated by a permeable membrane. [Permeable membrane- Which selectively does not permit the passage of dissolved solute particles, molecules, ions etc.]
  - Then flow of solvent takes place from dilute to concentrated sides due to osmosis. If, however a hydrostatic pressure in excess of osmotic pressure is applied on the

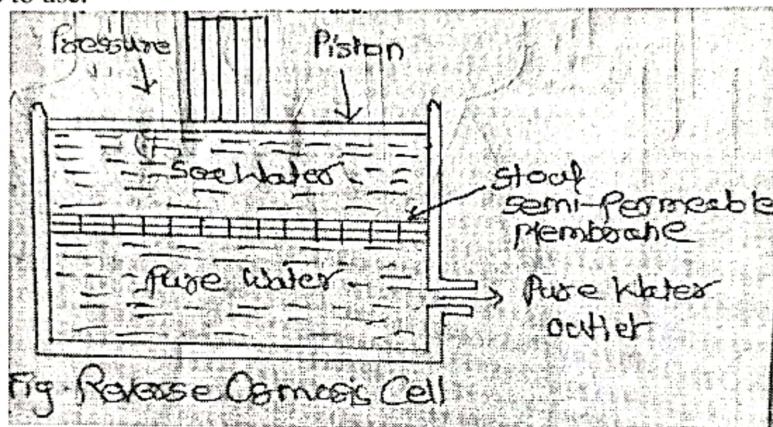


concentrated side, the solvent flow reverses, i.e. to dilute side across the membrane. This is principle of reverse osmosis.

- 5) Thus, in reverse osmosis (RO) method, pure solvent (water) is separated from its contaminates rather than removing contaminants from their after. This membrane filtration is sometimes also called super-filtration or hyper-filtration.

6) **Method:-**

- In this process, pressure is applied to the sea water or impure water (to be treated) to force its pure water out through the semi-permeable membrane leaving behind the dissolved solids. (both ionic as well as non-ionic) (see fig. below)
- The membrane consists of very thin film of cellulose acetate, affixed to either side of a perforated tube. Recently superior membranes made of polymethacrylate polymers have come to use.



7) **Advantages:-**

- Removal of ionic as well as non-ionic, colloidal and light molecular weight organic matter is possible.
- It removes colloidal silica, which is not removed by demineralization.
- The maintenance cost is almost entirely of the semi permeable membrane also life of membrane is quite high.
- Membrane can be replaced within few minutes.
- Method is of low cost, simple and also operating cost is minimum.

**Question Bank**

Q.1 Enlist different impurities present in natural water and suggest methods for minimizing it. 4M

**Or**

Enlist different impurities present in natural water? How are they removed? 4M

**Or**

What are the different impurities present in natural water? 4M

Q.2 Write note on Total Dissolved Solids (TDS) 4M

Q.3 What is acidity of water? Explain in details, 5M

Q.4 Write short note on 'Alkalinity of water sample'. 4M

Q.5 What is chloride content in water? Explain in detail, 5M

**Or**

Write note on chloride content in water. 4M

## Unit 2. Instrumental Methods of Chemical Analysis

### **Introduction:**

In early days mostly manual methods are available for analysis such as titrimetric methods etc. Now this analysis we can do easily & accurately using various instruments such as  $p^H$  meter, potentiometer, conductivity meter

There are two types of chemical analysis

#### A) Qualitative analysis:

The method in which what is present in organic & organic chemical species is detected, only qualitative determinations are done.

#### B) Quantitative analysis:

In this analysis what is & how much concentration/activity of chemicals species present is determined

**Instrumental Methods are now preferred over manual methods because of following advantages:**

- 1) A small amount of a sample is needed for analysis.
- 2) Fast result gained by using instrumental methods.
- 3) Complex mixture can be analyzed either with or without separation.
- 4) Sufficient reliability and accuracy of results are obtained by instrumental method.
- 5) When non-instrumental method is not possible, instrumental method is the only answer to the problem.
- 6) Under fixed condition we can reproduce the result.
- 7) New instrument become easy to handle & operate.
- 8) Most of instruments are now computable with printer & other devices. So data can be stored, printed & managed easily.

### **Disadvantages of instrumental Methods:**

- 1) In general, instrument methods are costly because of cost, maintenance and trained personal required for their handling.
- 2) The sensitivity and accuracy depends upon the type of instrument.
- 3) Specialized training for handling instrument is required.
- 4) There is frequent need of checking results with other methods.
- 5) In some cases, instrumental method may not be specific.

- **Spectrometry:** -

#### I) **Introduction:**

- 1) Photometry or spectrophotometer measure intensity as a function of colour or more specifically, the wavelength of light.
- 2) This device is used to determine the concentration of known solute in given solution by the application of Lambert's-Beer law which states that concentration of solute is proportional to absorbance.

- 3) They are commonly used in scientific fields such as chemistry, biochemistry, physics and molecular biology. Also in industries.

## II) Laws of spectrometry:

### A) Lambert's Law

**Statement-** “When beam of monochromatic light passes through a transparent medium the rate of decrease of intensity of light with the thickness of the absorbing medium is proportional to the intensity of incident light.”

**Proof: -**

According to statement,

$$-\frac{dI}{dt} \propto I$$

$$-\frac{dI}{dt} = kI$$

$$\frac{dI}{I} = -kdt \quad \dots \dots \dots 1$$

Where,  $I$  = Intensity of incident light of wavelength  $\lambda$ .

$dI$  = decrease of intensity when light passes through a medium of thickness  $dt$ .

$k$  = proportionality constant or absorption coefficient.

If  $I_0$  is intensity of light before entering the absorbing medium when  $t=0$ , the intensity of light  $I_t$  after passing through thickness  $t$  of the medium can be obtained by integrating **equation 1** between these limits.

$$I_0 \int^{I_t} dI/I = -k \int_{t=0}^{t=t} dt$$

$$I_0 [\log I]^{I_t} = -kt$$

$$\log I_t / I_0 = -kt$$

$$I_t / I_0 = e^{-kt}$$

$$I_t / I_0 = 10^{-kt/2.303}$$

$$I_t / I_0 = 10^{-at}$$

Where,  $a = k/ 2.303$ ,  $a$  is called as extinction coefficient.

### B) Lambert-Beer's Law (Beer's law)

**Statement-** “When beam of monochromatic light passes through a solution of absorbing substance the rate of decrease of intensity of light with the thickness of the absorbing solution is proportional to the intensity of incident light as well as concentration of solution.”

**Proof: -**

According to statement,

$$-\frac{dI}{dt} \propto Ic$$

$$-\frac{dI}{dt} = kIc$$

$$\frac{dI}{I} = -kcdt \quad \dots \dots \dots 1$$

Where,  $I$  = Intensity of incident light of wavelength  $\lambda$ .

$\Delta I$  = decrease of intensity when light passes through a absorbing solution of thickness  $dt$ .

$k$  = proportionality constant or absorption coefficient.

$c$  = concentration of solution in mol/dm<sup>3</sup>.

If  $I_0$  is intensity of light before entering the absorbing solution when  $t=0$ , the intensity of light  $I_t$  after passing through thickness  $t$  of the medium can be obtained by integrating equation 1 between these limits.

$$I_0 \int I_t dI/I = -kc \int_{t=0}^{t=t} dt$$

$$I_0 [\log I]^{I_t} = -kct$$

$$\log I_t / I_0 = -kct$$

$$I_t / I_0 = e^{-kct}$$

$$I_t / I_0 = 10^{-kct/2.303}$$

$$I_t / I_0 = 10^{-\epsilon ct}$$

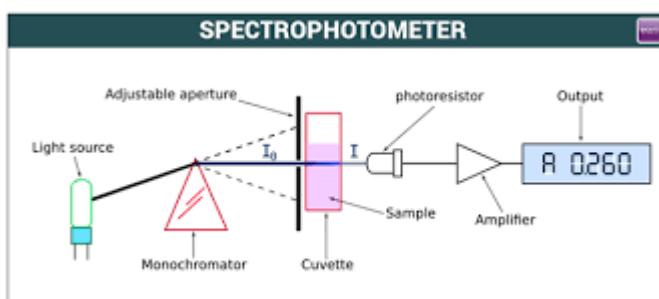
Where,  $\epsilon = k / 2.303$

$\epsilon$  is called as molar extinction coefficient.

### III) Single Beam spectrophotometer:

1) Single beam spectrophotometer measures the absolute light intensity.

2) Schematic Diagram:



3) Construction: -

It consists of following parts

- i) **Source:** - A light source usually an ordinary low- voltage filament is used. It has wavelength range (visible region) 400-700 nm.
  - ii) **Monochromatic:** - It is set of filters this select a light of proper wavelength falling on it and passes to sample cuvette.
  - iii) **Sample Cuvette:** - It holds sample whose concentration is to be determined.
  - iv) **Detector:** - Detector is generally a light sensing device like photo resistor or photodiode which measures the transmitted light.
  - v) **Read out Device:** - This reads the results usually these contains a monitor and printer.
- 4) **Working:** - In single beam spectrometer, light from source falls on Monochromator which select a spectrum of proper wavelength. This spectrum of proper wavelength falls

on cuvette containing a sample. Then transmitted light from cuvette is detected at detector and signal is processed in readout device.

### 5) Applications

Applications UV/Vis spectroscopy is routinely used in analytical chemistry for the quantitative determination of different analytes, such as transition metal ions, highly conjugated organic compounds, and biological macromolecules. Spectroscopic analysis is commonly carried out in solutions but solids and gases may also be studied.

- i) Solutions of transition metal ions can be colored (i.e., absorb visible light) because d electrons within the metal atoms can be excited from one electronic state to another. The colour of metal ion solutions is strongly affected by the presence of other species, such as certain anions or ligands. For instance, the colour of a dilute solution of copper sulfate is a very light blue; adding ammonia intensifies the colour and changes the wavelength of maximum absorption ( $\lambda_{\text{max}}$ ).
- ii) Organic compounds, especially those with a high degree of conjugation, also absorb light in the UV or visible regions of the electromagnetic spectrum. The solvents for these determinations are often water for water-soluble compounds, or ethanol for organic-soluble compounds. Solvent polarity and pH can affect the absorption spectrum of an organic compound.
- iii) UV/Vis can be applied to determine the kinetics or rate constant of a chemical reaction. The reaction, occurring in solution, must present color or brightness shifts from reactants to products in order to use UV/Vis for this application.
- iv) An equilibrium constant can also be calculated with UV/Vis spectroscopy. After determining optimal wavelengths for all species involved in equilibria, a reaction can be run to equilibrium, and the concentration of species determined from spectroscopy at various known wavelengths. The equilibrium constant can be calculated as  $K(\text{eq}) = [\text{Products}] / [\text{Reactants}]$ .
- v) Detection of impurities- The UV absorption Spectrophotometry is one of the best methods for identification of impurities on organic compounds.
- vi) Molecular weight determinations- Molecular weights of compounds can be measured spectrophotometrically provided suitable derivatives of these compounds could be prepared.

- **Chromatography: -**

- 1) It is a physical method of separation in which the components to be separated are distributed between two phases, one of which is stationary phase while the other (mobile phase) moves in a definite direction.
- 2) Literally ‘chromatography’ means ‘colour writing’.

**Types of chromatography: -**

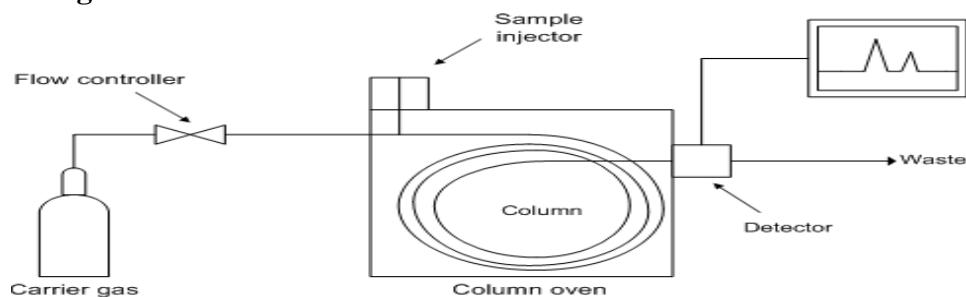
- A. Techniques by chromatographic bed shape

- a) Column Chromatography
- b) Planer Chromatography-i) Paper Chromatographyii) Thin Layer Chromatography
- B. Techniques by physical state of mobile phase
  - a) Gas Chromatography
  - b) Liquid Chromatography
- C. Techniques by separation mechanism
  - a) Ion Exchange Chromatography
  - b) Size Exclusion Chromatography

➤ **Gas Chromatography:**

Gas chromatography is also known as gas liquid chromatography in which separation of sample mixture is done by using gas (inert gas Helium) as mobile phase. Inert gas carries sample therefore it is also called as carrier gas.

**Schematic Diagram**



**Instrumentation: -**

- 1) **Auto sampler:** -Auto sampler provides the means to introduce a sample automatically into the inlets.
- 2) **Inlets:** -The common inlet provides the means to introduce a sample into continuous flow of carrier gas.
- 3) **Columns:** - Two types of columns are commonly used,
  - a) **Packed Columns:** - Column having 1.5-10 nm length and internal diameter of 2-4 mm. The tube is usually made of stainless steel or glass and contains a packing of finely divided, inert solid support material that is coated with a liquid or solid stationary phase.
  - b) **Capillary Column:** - It has very small internal diameter and length is in between 25-60 meters. The inner column walls are coated with the active material.
- 4) **Detectors:** - A number of detectors are used in gas chromatography. The most common are flame ionization detector (FID) and thermal conductivity detector (TCD). Both are sensitive to wide range of concentrations.

**Working: -**

In a GC analysis, a known volume of gaseous or liquid analyte is injected into the entrance of the column using micro syringe. As carrier gas sweeps the analyte molecules these molecules

are absorbed on side walls or by packing material. The rate of adsorption depends on molecules, stationary phase etc. The stream of molecules coming out of column is detected.

#### ➤ Applications of Gas Chromatography

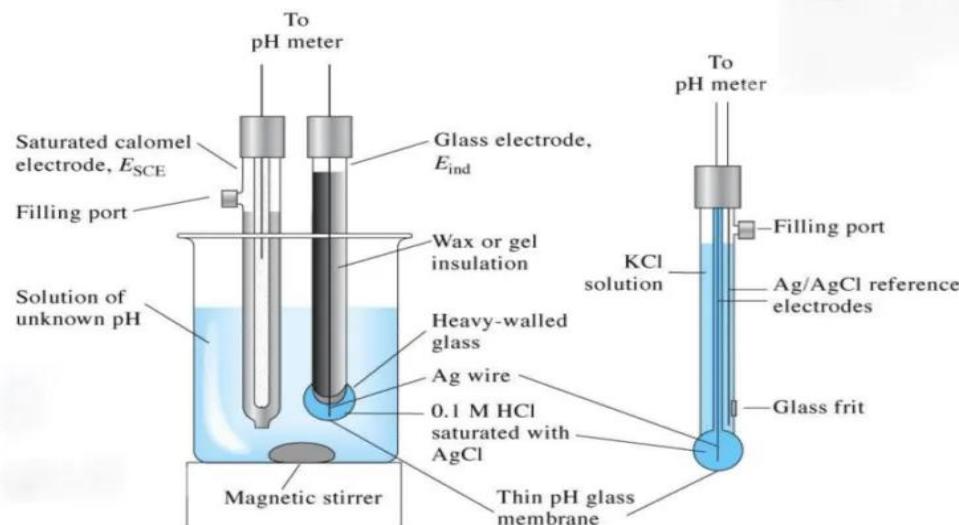
- 1) A specific application of GC is measuring a purity of particular substance, or separating components of a mixture. Also relative amounts of such components can also be determined.
- 2) In some cases, GC may help on identifying a compound and also can be used to prepare pure compounds from mixture.
- 3) To analyze content of chemical product.  
e.g. to assure quality of product in chemical industry, measuring toxic substance in soil, water or air.
- 4) Gas chromatographs connected to mass spectrometers which act as detector. It is also called as GC-MS. Further it can be connected to NMR (GC-MS-NMR) by following IR also (GC-MS-NMR-IR).
- 5) Qualitative analysis: - Generally chromatographic data is presented as graph (called chromatogram) of detector response at Y-axis against retention time at X-axis. In most of cases GC is connected which is capable of identifying the analyte represented by the peaks.
- 6) Quantitative analysis: - The area under the peak in chromatogram is proportional to the amount of analyte present in the sample. By calculating the area of the peak using the mathematical function of integration, the concentration of analyte in original sample can be determined.

### • pH Meter

A pH meter is used to measure the acidity and alkalinity of a solution, or a pH meter is an electronic device used for measuring the pH of any solution.

#### pH Meter Principle

It measures the voltage between the two electrodes. One is a glass electrode, and the other is a reference electrode. It displays the result of that voltage that is related to the corresponding pH value. Sometimes, if both electrodes are present, it is called the combination electrode, and they are inserted into the solution in which pH is to be tested. These two electrodes are immersed and, after immersing these electrodes in a solution. That  $H^+$  ion in the test solution exchange for other positively charged ions presents on the glass bulb. So there is an action between these plus ions of the solution and  $H^+$  ions or positively charged ions present on the glass bulb. The amplifier detects the difference in electric potential between the two electrodes. The contrast of these potentials is called the pH unit.



### Working of pH meter

The working of pH meter is based on Nernst equation. Nernst equation derives the relation between the electric voltage and ion concentration. The Nernst equation derived for  $H^+$  ion concentration is the basis of pH meter. The working principle of pH meter is the potentiometric. The pH meter consists of glass (also called as indicator electrode) and reference electrode. The glass electrode consists of glass membrane, which is sensitive to hydrogen ion concentration of test sample solution. And the glass electrode potential varies from sample to sample. The reference electrode is standard and has constant potential. The reference electrode does not respond to test sample solution. The pH meter measures and compares the potential difference between both glass and reference electrodes

### Construction Glass Electrode

The glass electrode is constructed from a special soft glass of high electrical conductance. It consists of a bulb that contains a solution of constant hydrogen ion concentration. Usually, the solution contains 0.1 N hydrochloric acid (HCl) in which a silver/silver chloride electrode is dipped. The bulb is then placed in a solution of unknown pH.

### components of pH instrument

1. Glass bulb: It is a sensor that senses the  $H^+$  ion concentration and it is made from special type of glass and membrane. The glass bulb consists of 0.1M HCl.
2. Internal electrode: It is the silver chloride electrode.
3. Internal solution: The silver chloride electrode is dipped in buffer solution of 0.1 mol/L KCl of pH 7.
4. Reference Electrode: It is also the silver chloride electrode.

5. Internal Solution: The reference electrode is also dipped in buffer solution of 0.1 mol/L KCL of pH 7.
6. Junction: It is made from ceramic junction also called as diaphragm that allows the contact of sample solution and reference electrolyte. It does not disturb the electric connection between both the electrodes.
7. Filling hole: It is used for refilling the electrolyte.
8. Electrode body: the body is from non-conductive glass or plastic.

### Applications of pH Measurement

The applications include:

- pH Measurement is very crucial in Agriculture industry for soil evaluation. Major crops require alkaline environment and hence pH Measurement becomes necessary.
- It is also used in Food industry especially for dairy products like cheese, curds, yogurts, etc.
- It becomes mandatory for chemical and pharmaceutical industries.
- It becomes a significant factor in the production of detergents.
- pH level monitoring is essential in water treatment plants and RO water purifiers.

### Advantages of pH Measurement

The advantages are:

- pH Measurement is inexpensive and robust.
- Pocket size pH Meters are user friendly.
- Readings are accurate and precise.

### Question Bank

Q.1 What are the advantages and disadvantages of instrumental methods of analysis? **5M**

**Or**

Write objective and advantages of instrumental methods of chemical analysis. **5M**

**Or**

What are the advantages of instrumental methods of chemical analysis over non-instrumental methods? **5M**

**Or**

Enlist Merits & demerits of instrumental methods **4M**

Q.2 Write short note on Lambert law. **4M**

Q.3 State and derive an equation for Beer's-Lambert's law. **4M**

**Or**

Write note on Beer-Lambert's law. **4M**

**Q.4 With schematic diagram, explain principle, construction and working of single beam spectrophotometer. **6M****

**Or**

**Explain the single beam spectrophotometer with schematic representation. **5M****

**Or**

**Explain instrumentation of single beam and spectrophotometer. **4M****

**Q.5 With schematic diagram, explain principle, instrumentation and working of GLC. **6M****

**Q.6 Enlist the advantages and applications of gas chromatography. **4M****

**Or**

**What are applications of gas chromatography? **4M****

**Q.7 With schematic diagram, explain principle, construction and working of PH meter .**