# CHAPTER – III

# EXPERIMENTAL & METHODS

# **CONTENTS**

- 3.1 GENERAL INTRODUCTION
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This chapter deals with experimental data of water analysis in terms of pH, hardness, total alkalinity, fluoride, phosphate, chloride, calcium, magnesium, and nitrate, C.O.D, B.O.D, dissolved oxygen (DO), total dissolved solid (T.D.S) etc. of two Talukas of Bharuch district with 15 stations in each Talukas. Data obtained in three seasons such as Winter, Monsoon, and Summer are given in chapter-4.

#### 3.1 GENERAL INTRODUCTION

This chapter deals with the collection of samples of Ground water from Ankleshwar and Bharuch Taluka of Bharuch district with 15 stations in each Taluka in three different seasons such as Winter, Monsoon, and Summer and their analysis.

As the rain fall is uneven from past few years, the potentials of small/seasonal rivers affect the ground water in different ways. This can make a large change in property of ground water, which could be temporary or permanent and hence the property of ground water turns poor.

Groundwater system is polluted by natural activities as well as human activities such as: Pollutants dumped on the surface of land comes in contact with percolating water of rain which make the soluble part dissolved in them and picks it up to the aquifer. The boisterous of surface water pollution in various levels of the nation has become so severe that if crucial ladders for reduction are not adopted surface water capitals may be injured.

Groundwater property depends on number of factors such as:

- 1. Number of hydrological factors: Variation in seasons
- 2. Number of physical factors: Type of soil, moisture content etc.
- 3. Number of chemical factors: Soil pollution by Industry and constitution of upper crust of soil.
- 4. Number of biological factors: Surrounding Plant Kingdome and animal Kingdome.

It is generally found that the groundwater has more dissolved constituents as compared to surface water as it is more in contact with different types of materials than surface water. As the constitution of soil varies from point to point, there is vast difference in constitution of groundwater. Potable water should ideally be free from toxic elements, dissolved minerals in specific limit; some heavy metals in specific limit for example

Cobalt, Copper, etc. The foremost serious problem is the increase in fluoride content in ground water leading to the disease called Fluorosis. Most of the peoples using ground water for the drinking purpose, so it's necessary to know about the quality of the ground water, suspended solids and impurities. Here, in present work we have selected two talukas, namely, Ankleshwar and Bharuch of Bharuch district, Gujarat with fifteen stations in each taluka with respect to three different seasons Winter, Summer and Monsoon.

#### 3.2 SAMPLING

Two different taluka of Bharuch district with 15 stations were selected for each taluka for the physicochemical analysis of ground water with respect to three different season such as Winter, Summer and Monsson. Quality of ground water was determined in terms of measurement of physico-chemical parameters such as concentration of different ions like chloride, fluoride, nitrate, calcium, sulphate and magnesium, pH, Chemical oxygen demand (COD), Biological oxygen demand (BOD), total alkalinity, dissolved oxygen (DO), and total dissolved solid (TDS). Water samples collected from tube well, open dug, hand pump etc. from the different stations namely Sarthan, Telva, Piludra, Umarwada, Jetali, Piprod, Avadar, Pardi Mokha, Sangpor, Kosamadi, Panoli, Kharod, Motali, Andala, Utiyadara, Adol, Amdada, AmLeshwar, Bambusar, Bhuva, Cholad, Dabhali, Ghodi, Haldar, Karela, Kelod, Osara, SamLod, Segva and Shahpura in 2.5 L precleaned glass sample bottles.

# 3.3 EXPERIMENTAL

# Total Dissolved Solid (TDS)/ Suspended Solid

**Principle: -** TDS is nothing but combination of dissolved solid of true solution and combined solution called pseudo solution.

**Suspended Solid:** - This type of the solid can be removed simply by the filtration using appropriate size filtration device and organic suspended solid can be simply removed by choosing appropriate filter paper and funnel.

**Dissolved Solid:** - Salts such, sulphate, nitrate, bicarbonates and chlorides of calcium and magnesium are crystalline can be removed by filtration using glass funnel and filter paper or upon boiling of the amounts of salts in liquid and calculating the liquid left behind. In majority of cases when samples are dried at temperature 102-105°C but in some of the cases, it may dried up to temperature 130-170°C results in to removal of few organic

compounds, Nitrate and Chlorides in addition to bicarbonates gets converted to carbonates that's why exact TDS determination is not possible.

#### Procedure: -

#### (1) Total Dissolved Solid (TDS)

For determination of total dissolved solid in the sample G4 sintered funnel was used for the removal of the organic impurities and other suspended solid followed by preparation of sample in the 100 mL previously cleaned beaker (pre heated at above 120°C temperature and suddenly cooled down to room temperature). Sample was evaporated using steam bath at temperature around 100°C till the constant weight was observed at least for three times in the oven. steam bath (102-105°C) was used till constant weight was recorded for 3 times followed by cooling at room temperature and weighing it calculation was done as per the formula given below

#### **Calculation:**

# (2) Total Suspended Solid (TSS)

To find out suspended solid in water determined by taking sample in G4 funnel preheated at temperature 120°C and filter it using What man filter paper followed collection of filtrates in the beaker. Solid collected in the form of residue on filter paper were washed with the double distilled water. The solid collected over the funnel/paper were washed with double distilled water and deride at 100°C temperature and weight out.

#### **Calculations:**

#### (3) Total Solid: -

To determine total solid in the water sample, water is collected in the previously dried beaker at 120°C temperature and was placed in oven at temperature 170°C till the constant weight is recorded followed by allowed to cool at room temperature in desiccator for long time. Sometimes solid dried at room temperature may absorb moisture due to hygroscopic its nature

#### **Calculation**

# pH Measurement

**Principle:** - Using these methods one can determine whether water is acidic or basic in nature. Mathematically, pH is determining by measuring concentration of hydronium ion.

$$pH = -\log[H_3O^+]$$

This assembly made up of two components such as glass and reference electrode: **Glass Electrode:** - It is type of voltmeter which is sensitive towards H<sup>+</sup> ion concentration. Key role of this electrode is to separate standard 0.1M HCl solution on inner side of glass electrode and sample solution on outer side of glass electrode using thin glass membrane. Electrode is made up of silver road coated with silver chloride which is represented by Ag/AgCl. Potential is depending on concentration of acid solution.

**Reference electrode:** - This electrode is nothing but silver metal is in contact with d silver chloride dipped in 0.1M KCl solution surrounded by glass tube having porous fiber to conduct electricity. Potential difference is created between potassium chloride and sample solution.

Note: for calibration, the buffer solution with pH 7.0 was used and tap water's pH was found between 6.5-7.0.

#### Procedure: -

First, the pH meter was started and kept for 5 minutes to stabilize and them followed by calibration using buffer solution of pH 4.0 and 9.2 (Acidic and basic respectively).

Temperature was kept constant throughout the experiments and the 10 H of water sample was measured after properly dippy the elects in the water sample.

# **Total Hardness**

Principle: - Water is hard due to presence of satts of calcium and magnesium. Presence of hardness is mark by slat formation while using as the coolant. Elimination of hardness can be possible by softening of water, ion exchange resin method, Electrolysis, etc. by removing hard ions, water become soft.

Complexometric method is available for determination of hardness in water sample using EDTA by formation of complex with Ca<sup>2+</sup> and Mg<sup>2+</sup> ions at basic pH using Eriochrome Black-T indicator. Color change appear from wine red to blue.

Fig. 3.3 Eriochrome Black-T [EBT]

#### **Procedure:**

Exact 50mL of water sample was taken in un 250 mL conical flask. 2-3 mL buffer solution of basic pH was added, followed by addition of Eriochrome black T as the indicator. The solution was stirred well and titrated it with 0.01M EDTA solution till the color change was observed from wine red to blue. The experiment was repeated for two more reading and noted the constant reading as the end point.

Calculation: Total Hardness

 $M_1V_1=M_2V_2$ 

Where,

M<sub>1</sub>= Concentration (Hardness) of water sample, V<sub>1</sub>= Volume of water taken

M<sub>2</sub>= Concentration (0.01M) of EDTA, V<sub>2</sub>= Volume of EDTA

Hardness due to Ca<sup>2+</sup>

**Principle:** Murexide (ammonium purpurate) indicator was used to determine presence of calcium in the mixture of calcium and magnesium using the complexometric titration with EDTA at pH greater than 11. At alkaline pH, magnesium is precipitated as the magnesium hydroxide and it does not interfere during the titration. Initially, pink color appears due to unstable complex formation of calcium with indicator followed by formation of new complex with EDTA which has purple color indicating the end point. The experiment was repeated for two more reading and the end point was determined.

Procedure: -

50mL of water sample was taken in the conical flask, add 2mL 1N sodium hydroxide solution was added at so that the pH becomes more alkali say 12. Now titrate the solution against 0.01M EDTA using murexide as the indicator. End point observed is color change from pink to purple.

Calculation: -

Calcium hardness in 
$$mg/mL = \frac{Volume \ of \ EDTA \ Consumed \ in \ mL \times 40.078}{Volume \ of \ water \ sample \ taken \ in \ mL}$$

Hardness due to Mg<sup>+2</sup>

**Principle:** - Whenever mixture of magnesium and calcium is present at that time there is no direct method available for the determination of only magnesium concentration but it can be determine using the following formula.

**Magnesium hardness in mg/mL = Total hardness - hardness due to Calcium in <math>mg/mL** 

# Cl- by Argentometric method

For any titrimetric method

- (a) reaction must be completed i.e. K<sub>titration</sub> should be larger.
- (b) Reaction must be fast

Precipitation reaction itself is the fast reaction process therefore for reaction of silver salt uses argentometric titration in which silver nitrate is used as the titrant. This method is applicable to analyze different negative ions which are capable of forming precipitates with  $Ag^+$  ion.

Silver is precipitated as the silver chloride and silver nitrate is taken in burette and when it is added to water sample, then precipitation of AgCl take place, here potassium chromate is used as the indicator.

$$Ag^+(Burette) + Cl^-(Sample) \rightarrow AgCl(\downarrow)$$
 White ppts,  
 $Ag^+(Burette) + CrO_4^2(Sample) \rightarrow Ag_2CrO_4(\downarrow)$  Red ppts



**Fig. 3.6** 

But whenever there is a free chloride ion in the medium the red colour of silver chromate disappear because of the following reaction:

Ag<sub>2</sub>CrO<sub>4</sub>(
$$\downarrow$$
) Red precipitates + 2Cl<sup>-</sup>(Sample)  $\rightarrow$  2AgCl( $\downarrow$ ) White precipitates + CrO<sub>4</sub><sup>2-</sup>

Hence at the end point of the reaction; no free chloride ions are available, the following reaction takes place:

$$2AgNO_3 + K_2CrO_4 \rightarrow Ag_2CrO_4(\downarrow)$$
 Red precipitates +  $2KNO_3$ 

#### **Process:**

**Blank Titration (V<sub>1</sub>):** 50 mL deionized double distilled water was taken in conical flask, 2 mL 2N Potassium chromate was added to it and titrated against 0.01N AgNO<sub>3</sub> solution taken in burette. Experiment was repeated to get constant reading as the end point, called  $V_1$  mL.

**Sample Titration (V<sub>2</sub>):** 50 mL sample water was taken in conical flask, and 2 mL 2N Potassium chromate was added to it and it against 0.01N AgNO<sub>3</sub> solution taken in burette. Experiment was repeated to get constant reading at the end point, called V<sub>2</sub> mL.

#### **Calculations:**

Required volume of AgNO<sub>3</sub> for consumption of  $Cl^-$  present in water sample =  $V_2$ - $V_1$  mL

 $= V_3 mL$ 

Concentration of Silver nitrate  $N_3 = 0.01N$ 

Volume of water sample taken = $V_4$ =50 mL Concentration of Cl<sup>-</sup>( $N_4$ ): can be determine as following

Cl<sup>-X</sup> concentration in g/mL=N<sub>4</sub> 
$$\frac{N_3V_3\times35.49}{V_4\times1000}$$

# Determination of sulphate ion by spectrophotometric method

**Principle:** - First standard plot of absorption of sulphate ion from known concentration of BaSO<sub>4</sub> prepared and same was carried out for the sulphate ion present in the water sample. By comparing both the plots one can measure the concentration of sulphate ions present in the water sample.

#### **Reagents:**

Reagents: In a 250 mL measuring flask 25 mL Glycerol, 15 mL conc HCl and 50 mL isopropyl alcohol and 35 g NaCl. mL was taken The volume of flask was made 250mLwith distilled water.

Stand. Solution of Sulphate ion 1mL = 1mg Sulphate ion: It was prepared by dissolving 1.479 g anhydrous sodium sulphate in to 1000 mL distilled water.

# Preparation of standard solutions, sample solution and blank solution:

	Flask Number				
1	2	3	4	5	6
0.0 mL	10.0 mL	20.0 mL	30.0 mL	40.0 mL	20.0 mL
standard	standard	standard	standard	standard	sample
solution	solution	solution	solution	solution	solution
5 mL reagent	5 mL	5 mL	5 mL	5 mL	5 mL
	reagent	reagent	reagent	reagent	reagent
10mL BaCl <sub>2</sub>	10mL	10mL	10mL	10mL	10mL
solution	BaCl	BaCl	BaCl	BaCl	BaCl
	solution	solution	solution	solution	solution
85 mL distilled water	75 mL	65 mL	55 mL	45 mL	55 mL
	distilled	distilled	distilled	distilled	distilled
	water	water	water	water	water

#### **Process:**

Length wave of spectrophotometer was set to 420 nm. Instrument was calibrated by taking blank solution from flask number-1 in to photo-cell. The it was set to zero by measuring the absorption of both sample and standard solutions.

The absorption of standard and sample solution was plotted against concentration of sulphate ions. From the graph, one can know the required concentration of sulphate ions corresponding to absorption.

# Determination of Nitrate ion by spectrophotometric method

# **Principle:**

This method is only applicable when water sample contains low nitrate level. In this method is based on back estimation as follow:

Length wave of spectrophotometer was set to 220 nm and absorption was measured due to presence of organic matter and nitrate ion. The same measurement was made at wave length 270 nm which resulted in to the measurement of absorption only due to organic matter and not because of nitrate ions therefore, by taking difference of these two results

gives the absorption due to nitrate ions is obtained.

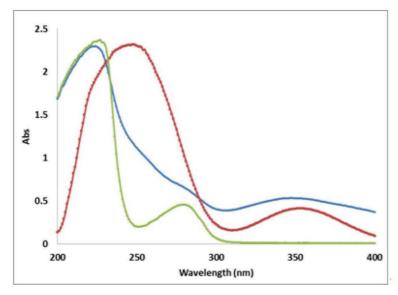


Fig. 3.7

Sample absorption curve for Nitrate determination.

# Reagents: -

Double distilled water without having nitrate ions.

*Stock nitrate solution:* 0.7218 g anhy potassium nitrate was dissolved in deionized water and the total volume was made to 100 mL.

*Hydrochloric acid:* 1N HCl.

#### **Procedure:**

**Standard curve preparation:** Series of solutions of nitrate ions with concentration in the range of 0-0.35  $\mu$ g/ mL. were prepared Also mL HCl was added to each solution.

**Sample preparation:** 50 mL water sample was taken and 1mL HCl was added to it.

Measurement of Absorption: - First reference solution was taken in the photometric cell and the absorption (O.D.) was measured at wave length 220 nm for absorption due to nitrate plus organic matter and at 270 nm for absorption due to organic matter only. Nitrate ions concentration was obtained by subtracting absorption at wave length 220 nm from the absorption at wave length 270 nm. Data obtained plotted as absorption vs concentration of nitrate ions straight line was obtained corresponding to water sample's absorption and it was extra plotted on x-axis to get concentration of nitrate ion present in the water sample.

# Determination of Fluoride ion by spectrophotometric method

*Principle:* - F<sup>-</sup> is generally very common in all water samples, but when it is present in but lower than this limit may causes dental problem such as dental caries but higher level (greater than 4 mg/L) result in to the fluorosis disease. This disease is very common in the various parts of India in which fluoride level is more than 10 mg/L.

Fluoride detection is done by decreasing in color intensity of zirconium ion resultant to colorless complex formation. (Absorbance was measured at 570 nm)

Fig. 3.8 Reaction between fluoride ion

#### Reagents: -

Water: Deionized double distilled water.

**Standard solution preparation:** - **0**.221g Sodium fluoride dissolved in 1 Liter of water and then again diluted by facing 100 mL of this solution water 1 Liter using water called 0.01 mg/L.

**SPADNS solution:** - 0.960g SPADNS taken in 500 mL water.

**Reference cell solution:** Take 10 mL of SPADNS solution in to 100 mL of water and 10 mL of Conc HCl solution.

SPADNS mixed reagent: 0.135 g ZrCl<sub>4</sub> 8H<sub>2</sub>O was added to 350 mL conc HCl and diluted this solution to 500 mL using distilled water.

**Process:** Various standard solution was made by taking 0, 2, 4, 6, 8, 12 and 14 mL stock solution in to 100 mL volumetric flask and was made to 100 mL using distilled water.

Then, 50 mL of each solution was taken in separate flask and 10 mL SPADNS was added reagent to each flask so the concentration of fluoride ion become 0, 0.2, 0.4, 0.6, 0.8, 1.0, 1.2 and 1.4  $\mu$ g/mL kept the solution was for 30 minutes at equal temperature.

# **Determination of Total Alkalinity by Titrimetric Method**

It is an important parameter which control potable and waste water. Alkalinity of water is due to presence of bicarbonate, hydroxide, carbonates, phosphate etc.

**Principle:** Alkalinity is measured for the neutralizing reaction with strong acid of known volume and concentration. Titration will give result of consumption of sulphuric acid and on the basis of that alkalinity is calculated.

Titration of samples were performed at acidic pH say 4.5 where indicator phenolphthalein remains colorless and then after adding base indicator gives pink color at pH of alkali range of 8.3.

# Reagents:

Water: Deionized double distilled water, 0.02N sulphuric acid was used

**Phenolphthalein Indicator:** 1 percent phenolphthalein in alcohol was used as the indicator.

Fig. 3.10

**Mix Indicator:** 0.02 g methyl red and 0.1 g bromocresol green were taken in 100 mL alcohol (95 %) and was kept in air tight container as the alcohol is volatile.

**Bromocresol** green

**Methyl Red** 

Fig. 3.11

# Process: -

100 mL water sample in to conical flask, add 2-3 drops of phenolphthalein indicator was added to it and titrate entire solution against 0.02 N H<sub>2</sub>SO<sub>4</sub> solution at the end point color change from pink to colorless, (called **Va** mL).



Fig. 3.12

In above same flask 2 drops of mixed indicator was added to mixture and titrated it against 0.02 N till the color change was observed from blue to red. Final end point reading called **Vb** mL.



Fig. 3.12

Calculation: Total Hardness

 $N_1V_1=N_2V_2$ 

Alkalinity due to hydroxide ion, Where

N<sub>1</sub>= Normality of H<sub>2</sub>SO<sub>4</sub>

 $V_1$ = Volume of  $H_2SO_4$  consumed (called  $V_a$  mL)  $N_2$ = Normality of hydroxide ion in water sample,  $V_2$ = Volume of water sample

Alkalinity due to other ions ion, Where: -

N<sub>1</sub>= Normality of H<sub>2</sub>SO<sub>4</sub>

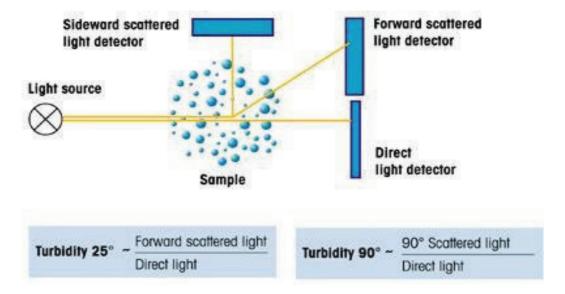
V<sub>1</sub>= Volume of H<sub>2</sub>SO<sub>4</sub> consumed (called V<sub>b</sub> mL)

 $N_2$ = Normality of carbonate/bicarbonate ions in water sample  $V_2$ = Volume of water sample

Results from above data is multiplied with their molecular weight. For hydroxide multiplied with 40 mol. Wt. of NaOH and for carbonate and bicarbonate multiplied with 100. As a result, alkalinity comes in mg/L. By addition of both, is obtained total alkalinity.

# **Turbidity determination by Turbidity meter**

*Principle:* Measurement of clarity of water sample is not directly possible but it is possible by measurement of transparency in the medium. Number of methods are available for determination of turbidity but one of the important methods is using turbidity meter. Turbidity is due to suspended organic and inorganic waste present in water sample.



Turbidity measured at 90° is called NTU

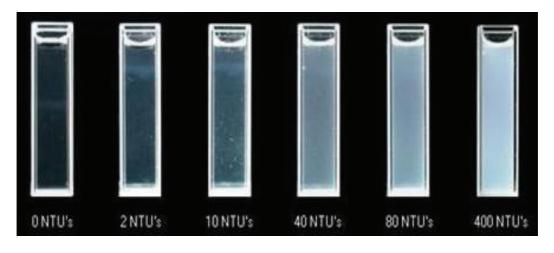


Fig. 3.13

#### **Process:**

Turbidity meter is calibrated with standard solution of 0.02 N NTU. Here, NTU stands for Nephelomatric Turbid is unit, which is a measure of turbid is in water samples is due to suspended solids. The relation between suspended solid and NTU is mg L<sup>-1</sup> suspended solid equipment to 3NTU according to Who Standard the turbitidity of drinking water must be less then 5NTU

Before measurement of turbidity, it is necessary that water attains at room temperature because, at lower temperature, turbidity increases.

Sample was inserted in the cuvette, it was allowed to get rid of air bubbles obtained during sample procedure. Then it was mixed to make homogeneous by micropipette.

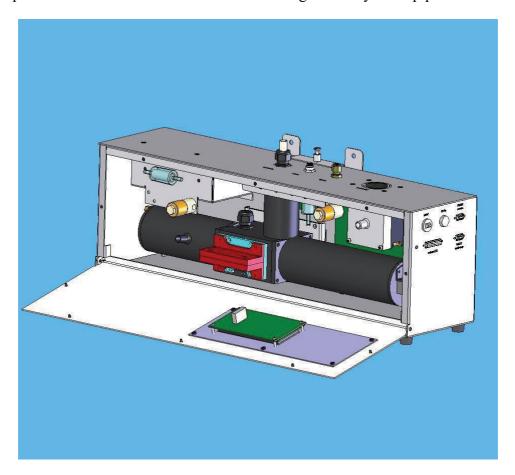


Fig. 3.14

After all this completed, outer surface was cleaned with the filter paper and turbidity measurement was made. Three consecutive readings were taken and the average value was obtained.

# Determination of dissolve oxygen by titrimetric methods

**Principle:** - Oxygen atom is present in the formula of water.in pure water, more oxygen gets dissolved in it. This is possible by removing carbon dioxide and hydrogen sulphide gas. We know that anything present in excess is poisonous, water with high concentration of dissolved oxygen cannot be used as the coolant in industry, because of its corrosion property. For titrimetric analysis, iodometric method is used.

#### Reagents: -

# Manganese sulphate solution: -

64 g Manganese sulphate was dissolved in water and the solution was made to 1000 mL using distilled water.

**Potassium iodide and Alkali solution:** - 150 g KI, 700 g KOH and 3 g NaN<sub>3</sub> was taken in 300 mL water and uniformLy mixed well and kept in cooled place under closed vessels.

Sulfuric acid (3:1): Take 750 mL H<sub>2</sub>SO<sub>4</sub> and 250 mL distilled water were taken and followed by cooling the solution at room temperature and diluted it to 1000 mL using distilled water.

**Sodium Thiosulfate Standard Solution (0.01N):** Standard solution of sodium thiosulphate was prepared by dissolving the 25 g of it in 1 liter of water. Standardization of this solution was done using known method.

**Starch Solution (Indicator):** 2% starch solution was prepared by taking 2 g starch in 100 mL water by heating it and allow to cool at room temperature.

#### **Process:**

A long tube, which is longer than BOD bottle (300 mL), was taken and this tube was placed in the sample bottle at one end and the other end was connected with sample filled and is was cap red such that there is no air bubbles left. Now 2 mL alkali solution and 2 mL manganese sulphate solution were added so that tip touches the surface of the solution without removing any air bubbles. The cap was replace again and content was mixed and kept stand still the precipitates settle down so that 2 mL 3:1 solution of sulphuric acid can be added without removing ppts. Same procedure was applied for KI and manganese sulphate. Reaction bottle is heated for agitation of ppts. Reaction time for entire procedure is 15 minutes. Now from this solution, 200 mL was taken and titrated against 0.01N

sodium thiosulphate solution which change in the color to blue at the end point.

# Calculation:

Dissolve Oxygen (mg/L) = 
$$\frac{A \times N \times 8000}{B}$$

Where,

A = burette reading

N = 0.01 i.e. normality of sodium thiosulphate, B = volume of sample taken in mL

Calculation for 200 mL sample taken from 300 mL is as follow:

$$B = \frac{200 \times 300}{300 - Y}$$

Where, y = 4 (i.e. 2mL alkaline KI and 2 mL 3:1 H<sub>2</sub>SO<sub>4</sub>)

# **Determination of Chemical oxygen demand (COD)**

# Principle:-

Chemical oxygen demand is the amount of oxigin required to oxidize the organic matter present in water and it is specific oxidizing agent present in water and the amount of it needed from outside for the experimental conditions. There are number of methods available for determination of COD, but open reflux method is widely used in industry, where large quantity of sample needed. (COD Value 50 mg/mL). Determination of COD given quintively, the pollution leaved of water due to organies. Higher value of COD indicates more pollution of organic matter in water.



Fig. 3.15

# Reagents: -

Standard  $K_2Cr_2O_7$  Solution: Dissolve 12.25 g potassium dichromate was dissolved in to distilled water and was made to 1liter Results 0.25N solution.

Sulphuric Acid: Conc H<sub>2</sub>SO<sub>4</sub> was used

*Ferrous ammonium sulphate (FAS):-* 39 g FAS was added to 20 mL conc H<sub>2</sub>SO<sub>4</sub> to prepare slurry and diluted to 500 mL distilled water to make clear solution followed by further dilution to 1 liter using distilled water.

*Ferroin Indicator:* This indicator was prepared by taking 0.695 g ferrous sulphate hepta hydrate and 1.485 g phenonthroline monohydrate in 100 mL water.

Mercuric Sulfate: solid form HgSO4 was used

#### Process:



Fig. 3.16

20 mL water sample was taken in 250 mL RBF fitted with refluxed condenser, 10 mL of prepared 0.25N potassium dichromate and 30 mL conc sulphuric acid were added and allowed to stand after each addition. 400 m mercuric sulphate was added to it and refluxed for 2 hr and the condenser was washed with distilled water. The entire mass was transferred in to 250 mL conical flask and diluted to 150 mL followed by titration of it against FAS solution (taken in burette) using ferrocin as the indicator. At the end point, color change from blues green to reddish brown. In the same way, the blank titration was performed without taking sample. COD was calculated as:

$$COD (mg/L) = \frac{(Sample-Blank reading) \times Normality of FAS \times 8000}{Volume of sample taken}$$

# **Determination of Biological Oxygen Demand (BOD)**

BOD is nothing but it is the quantity of dissolved oxygen required by aerobic biological organism to dissociate organics present in water sample at definite temperature for a fixed duration of time. And saw it is the concentration of oxygen supplied by microorganism. Aerobic bacteria use organic matter as their food. BOD value are effective parameter to know the pollution leaved and hence quiets of water. It is used as the index for was the water treatment plants. COD and BOD both are indication of organic pollution in water, but COD is less specific were BOD is high, DO value is less and the organism which required more DO, cannot survive.

# Principle: -

To evaluate burden on the efficient treatment plants (ETP plant), principle involve is that the back fall process may involve 5 days' time period, as a result, content which was use the oxygen called total biological oxygen demand. BOD5 term used for 5 days which is the time period required for the entire process. Treatment of plants are available in determination of uses of areal oxygen. Such type of organisms use areal oxygen. In the digestion process, the nitrogen compound is converted in to ammonia and CO<sub>2</sub>.

# Reagents: -

**Phosphate buffer:** 8.5 g KH<sub>2</sub>PO<sub>4</sub> + 21.75 g K<sub>2</sub>HPO<sub>4</sub>, 1.7 g NH<sub>4</sub>Cl 33.4 g, Na<sub>2</sub>HPO<sub>4</sub>•7H<sub>2</sub>O, were melt in 500 mL grade substance and diluted to 1000 mL by reagent score water. pH of solution was kept 7.2 & stored at lower temperature below 3 degrees Celsius.

*Magnesium sulfate solution:* 22.5 g MgSO<sub>4</sub>•7H<sub>2</sub>0 was dissolved in minimum amount of distilled water and then diluted 1 liter using same.

Calcium chloride solution: 27.5 g CaCl<sub>2</sub> was dissolved in minimum reagent and diluted to 1 liter using distilled with water.

*Ferric Chloride solution:* 0.25 g Ferric chloride was dissolved in minimum amount of reagent and diluted to 1 liter using distilled water.



Fig. 3.17

#### **Process:**

The pH of sample solution of water sample was kept from 6.5 to 7.5 and it was taken for determination of BOD. If pH value is not in specified required range, then one can set it using  $1N\ H_2SO_4$  or  $1N\ NaOH$ . Water sample is diluted to provide measurable BOD range. Deficiency of mineral and change in pH may lower the pH value. Hach BOD nutrient buffer pillows were used for precipitation of all BOD in the experiment specified by American Public health association (APHA) and environment protection agency (EPA). To ensure maximum level of oxygen it can aerate with 2  $\mu$ m in line filter for 20 minutes.

All bottles were made air tight and no extra water was allowed to come out from bottle by overflow. Poly propylene cap was used for the bottle. Surgical gum was used to remove BOD hooked in thin water.

For barometric pressure checking, the meter calibrated before use for every day analysis. DO was determined for blank solution also. Sample and blank solutions are kept at  $20 \pm 1$ °C for 5 days in the incubator. Check the daily added aquatic to closer if required.

# Calculations:

$$BOD (mg/L) = \frac{(Initial DO - DO_5) \times Dilution factor}{(Bottle volume 300mL)}$$

$$Sample Volume$$

Dilution factor = Dilution factor is the ration of the volume of diluted sample to volume of the undiluted sample

