UNIT - 5

ANALYSIS OF WATER

Turbidity:

- Turbidity is caused by particles suspended or dissolved in water that scatter light making the water appear cloudy or murky. Particulate matter can include sediment - especially clay and silt, fine organic and inorganic matter, soluble colored organic compounds, algae, and other microscopic organisms.
- Turbidity is the measure of relative clarity of a liquid. It is an optical characteristic of water and is a measurement of the amount of light that is scattered by material in the water when a light is shined through the water sample. The higher the intensity of scattered light, the higher the turbidity.
- NTU stands for <u>Nephelometric Turbidity unit</u>, i.e. the unit used to measure the turbidity of a fluid or the presence of suspended particles in water. The higher the concentration of suspended solids in the water is, the dirtier it looks and the higher the turbidity is.
- The good turbidity value of water is 0.1NTU or Below 0.1 NTU.
- Measuring turbidity is important when measuring the quality of water.

Turbidity is used to indicate the presence of pathogens, bacteria, and other contaminants such as lead and mercury which are harmful to both aquatic life and human health. This is why turbidity is measured in drinking water and wastewater systems.

DETERMINATION OF TURBIDITY OF WATER:

- The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity.
- Turbidity is the specialized term referring to the cloudiness of a solution and it is a subjective characteristic that is imparted by solid particles obstructing the transmittance of light passing through a water sample.
- Turbidity regularly demonstrates the presence of suspended solids like earth material, silt, clay, organic matter green growth, and different microorganisms.

EXPERIMENT (With Nepheloturbidity Meter):

Aim:

To determine the turbidity of the given sample of water by using the Nephelo turbidity meter.

Apparatus:

Nephrol turbidity meter, glass tubes, beakers.

Procedure:

- Turbidity is dependent on the comparison of the intensity of light scattered by the sample under characterized conditions with the power of the light scattered by a standard reference suspension under similar conditions.
- The turbidity of the sample is thus estimated from the amount of light scattered by the sample taking a reference with standard turbidity suspension. The higher the power of scattered light the more the turbidity.
- The unit of turbidity is expressed on NTU (Nephelometric Turbidity Unit).

Reagents:

1. Turbidity-free water:-

Generally, Distilled water is considered turbidity-free water for any test.

2. Stock turbidity solutions

Solution 1:-

For making a solution dissolve 1.0 grams of hydrazine sulfate $(NH_2)_2.H_2So_4$ in distilled water and dilute it to 100 ml in a makeup flask.

Solution 2:-

For the 2nd Solution Dissolve10.0grams hexamethylenetetramine

 $(CH_2)_6N_4$ in distilled water and dilute it to 100ml.

Solution 3:-Now, Mix solutions 1 and 2 in a 100ml flask each 5ml, and left it to stand for at least 24 hs., then dilute it to 100ml and mix thoroughly. The turbidity of this solution is 400 NTU.

Standard Turbidity Solution:-

For making a standard turbidity solution take 10.0ml of solution 3 in 100ml to make up the flask and dilute it to 100ml. with turbid–free water. The turbidity of this suspension is 40 NTU.

The procedure of Turbidity of Water Test:

a) Calibration of Nephelometer

1.Select the proper range of NTU on the Nephelometer.

2.Fill distilled water sample in a test tube and set the Nephelometer

reading to zero by using the knobs provided for the zero setting.

3.Now, fill the standard solution of 40 NTU in a test tube and set the reading to 40 NTU by adjusting the Nephelemeter reading to 40 NTU using a calibration knob.

b) Determination of turbidity of sample water

Turbidity less than 40 units:

- 1. It is **essential** to allow **samples** to come to **room** temperature **before** analysis.Mixthe **sample** to **thoroug hly** disperse the **solids**.
- 2. Wait until air bubbles disappear then pour the sample into the turbidity meter tube. Now, read the turbidity value in NTU directly from the instrument display or from the appropriate calibration curve.

Turbidity exceeding 40 units:

 In case of turbidity of the sample exceeds 40 units, and then add one or more same volume of distilled water or turbidityfree water in the turbid sample until the turbidity falls below 40 units. The turbidity of the test sample is calculated from the **turbidity** of the diluted sample and the **dilution factor.**

For example:If 5 same **volumes** of **distilled water** were **added** to the turbid sample, and

Where, A = Turbidity of the diluted sample, B = Volume of a diluted sample (ml) C = Sample volume taken for dilution. the **diluted** sample showed turbidity of **30 units**, then the turbidity of the **original sample** was **180 units**.

Nephelometric Turbidity Units (NTU) = A(B + C) / C

DISSOLVED SOLIDS

Dissolved solids" refer to **any minerals, salts, metals, cations or anions dissolved in water**. Total dissolved solids (TDS) comprise inorganic salts, principally calcium, magnesium, potassium, sodium, bicarbonates, chlorides, and sulfates and some small amounts of organic matter that are dissolved in water. Though an elevated TDS level can affect the taste of your water, **it is not usually harmful to human health**. However, readings above 500 ppm require further investigation for toxic particles and heavy metals, and readings above 1000 ppm are considered unsafe for human consumption. TDS in drinking-water originate from natural sources, sewage, urban run-off, industrial wastewater, and chemicals used in the water treatment process, and the nature of the piping or hardware used to convey the water, i.e., the plumbing.

The units for TDS usually are expressed as **milligrams per liter** (**mg/l**), which is the same as parts per million (ppm). Some meters show TDS as parts per thousand (ppt), which is equal to 1,000 ppm. The EC is a proxy measurement to determine the TDS in water.

DETERMINATION OF TOTAL DISSOLVED SOLIDS IN WATER:

Aim:

Determine the Total dissolved solids (TDS) of given water samples.

Introduction:

Water is a good solvent and picks up impurities easily. Pure water - tasteless, colorless, and odorless is often called the universal solvent. Dissolved solids" refer to any minerals, salts, metals, cations or anions dissolved in water. Total dissolved solids (TDS) comprise inorganic salts (principally calcium, magnesium, potassium, sodium, bicarbonates, chlorides, and sulfates) and some small amounts of organic matter that are dissolved in water.

In general, the total dissolved solids concentration is the sum of the cations (positively charged) and anions (negatively charged) ions in the water. Therefore, the total dissolved solids test provides a qualitative measure of the amount of dissolved ions but does not tell us the nature or ion relationships An elevated total dissolved solids (TDS) concentration is not a health hazard. The TDS concentration is a secondary drinking water standard and, therefore, is regulated because it is more of an aesthetic rather than a health hazard.

An elevated TDS indicates the following:

1)The concentration of the dissolved ions may cause the water to be corrosive, salty or brackish taste, result in scale formation, and interfere and decrease efficiency of hot water heaters.

2)Many contain elevated levels of ions that are above the Primary or Secondary Drinking Water Standards, such as an elevated level of nitrate, arsenic, aluminum, copper, lead, etc.

Requirements:

Water sample to be tested Evaporating Dish/ Ceramic Dish Desiccator Whatman Filter paper Electric balance machine

Procedure:

1. Filter your water sample through a Whatman Filter paper.

2. Collect the filtrate (liquid) and rinse water in a flask.

3. Take the weight of empty container (ceramic dish/ evaporating Dish). Make sure the container should be dried.

4. Add the filtrate to the container and allow the sample to stay in the oven at 103°C for 24 hours. If possible, increase the temperature of the drying oven to 180°C and allow the sample to dry for up to 8 hours.

5. Remove the container - Remember it is very hot. After removing from the drying oven, the sample should be placed in a desiccator to cool in a dry air environment for at least 3 to 4 hours.

6. After the container cools, reweigh the container at least three times.

7. Subtract the initial weight (in grams) of the empty container from the weight of the container with the dried residue to obtain the increase in weight.

Then do the following:

A-Weight of clean dried container (gm)

B-Weight of container and residue(gm)

C- Volume of Sample (ml)

Concentration $(mg/L) = ((B - A)/C)^* (1000 mg/g) * (1000 ml/L)$

For example:

A=100.0001gm B=100.0020gm C=100ml Concentration (mg/L) = ((100.0220 - 100.0001)/ 100) * 1000 * 1000 = 219 m

Precautions:

 If the sample cooled in a moist environment, the sample would increase in weightbecause of the addition of water vapor from the air.
Remember the sample is very hot and can melt plastic.

3. Do not touch container with bare hands.

HARDNESS OF WATER

The simple definition of water hardness is **the amount of dissolved calcium and magnesium in the water**. Hard water is high in dissolved minerals, largely calcium and magnesium. You may have felt the effects of hard water, literally, the last time you washed your hands.

TYPES:

Hardness of water can be of two types **temporary and permanent hardness**. Temporary hard water contains only bicarbonates of magnesium and calcium. It can be removed by boiling. Permanent hard water contains chlorides and sulfates of calcium and magnesium. **TOTAL HARDNESS OF WATER:**Total hardness is **the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter (mg/L)**.

You can determine your water's hardness based on these concentrations of calcium carbonate:

below 75 mg/L - is generally considered soft.

76 to 150 mg/L - moderately hard.

Determination of Total hardness of water by EDTA method:

Aim: Determine the total hardness of given water samples.

Introduction:

Hardness in water is due to the presence of dissolved salts of calcium and magnesium. It is unfit for drinking, bathing, washing and it also forms scales in boilers. Hence it is necessary to estimate the amount of hardness producing substances present in the water sample. Once it is estimated, the amount of chemicals required for the treatment of water can be calculated. The estimation of hardness is based on complexometric titration. Hardness of water is determined by titrating with a standard solution of ethylene diamine tetra acetic acid (EDTA) which is a complexing agent. Since EDTA is insoluble in water, the disodium salt of EDTA is taken for this experiment. EDTA can form four or six coordination bonds with a metal ion. Two type of hardness is present in water first is temporary hardness and second is permanent hardness. Temporary hardness is due to the presence of bicarbonates of calcium and magnesium ions.

It can be easily removed by boiling. Permanent hardness is due to the presence of chlorides and sulphates of calcium and magnesium ions. This type of hardness cannot be removed by boiling.

Requirements:

Water sample Burette 25-30ml Glass funnel Pipette 1ml Flask Dropper Measuring cylinder **Reagents:**

EDTA, Eriochrome Black-T, NH2CL, Ammonia Buffer, Magnesium Carbonate, 90% ethyl alcohol, Distilled water.

Reagent preparation:

1. EDTA solution: 4gm EDTA and 0.1gm magnesium bicarbonate dissolve in 800 ml distilled water.

2. Eriochrome Black-T: 0.4gm Ericrome Black T, 4.5 gm

hydroxylmine hydrochloride add in 100ml 95% ethyl alcohol.

3. Ammonia Buffer:

Stock A: 16.9gm of NH4CL in 143ml of conc. NH4OH,

Stock B: 1.25gm magnesium salt of EDTA dissolve in 50 ml distilled water.

Mix both stock solutions and dilute to 250ml with Distilled water. Dilute 10ml of the solution to 100ml with Distilled Water.

Procedure:

1. The burette is filled with standard EDTA solution to the zero level.

2. Take 50ml sample water in flask. If sample having high Calcium content then take smaller volume and dilute to 50ml.

3. Add 1ml Ammonia buffer.

4. Add 5 to 6 drop of Ericrome black – T indicator. The solution turns into wine red colour.

5. Note the initial reading.

6. Titrate the content against EDTA solution. At the end point colour change from wine red to blue colour.

7. Note the final reading and record it. Repeat the process till we get concordant value.

8. Take 50ml sample in another flask and boiled it. (Add distilled water to get final volume of water.)

9. Repeat step 3-7.

Calculations:

Total hardness of water mg/L (CaCO3 Scale) = ml of EDTA used (unboiled) *103 /ml of sample Permanent hardness of water mg/L (CaCO3 Scale) = ml of EDTA used (boiled) *103 /ml of sample

Temporary hardness of water mg/L (CaCO3 Scale) = Total hardness of water - Permanent hardness of water.

Result:

The collected water sample contains Total hardness = ____ppm

Permanent hardness = ____ppm

Temporary hardness = ____ppm

ALKALINITY

Definition :

"The buffering capacity of a water body; a measure of the ability of the water body to neutralize acids and bases and thus maintain a fairly stable pH level". It has a pH in the range of 8.0-9.0. *The good level of alkalinity is 20-200mg/L.

Cause:

• Alkaline nature in water is developed due to **the presence of impurities such as sodium carbonate, potassium bi carbonate or potassium carbonate**.

Uses:

1.Alkaline water has a higher pH level than that of plain tap water. So proponents say that it can neutralize acid in your bloodstream.

2.Alkaline water can help prevent disease, such as cancer and heart diseases.

3.Higher alkalinity levels in surface water will **buffer acid rain and other acid wastes, preventing pH changes that are harmful to aquatic life**. Alkalinity is also in important considering the treatment of wastewater and drinking water because it influences cleaning processes.

DETERMINATION OF ALKALINITY OF WATER

Aim: Determine the Alkalinity of given water samples.

Introduction: The alkalinity of the water is a measure of its capacity to neutralize acids. The alkalinity of natural waters is due primarily to the salts of week acids. Bicarbonates represent the major form of alkalinity. Alkalinity can be expressed as follows:

Alkalinity (mol/L) =
$$[HCO_3^{-1}] + 2 [CO_3^{2^{-1}}] + [OH^{-1}] - [H^{+1}]$$

Alkalinity is significant in many uses and treatments of natural waters and wastewaters. As alkalinity of many surface waters constitute of carbonates, bicarbonate and hydroxide contents, it is assumed to be an indicator of these constituents as well. Alkalinity in excess of alkaline earth metal concentrations is significant in determining the suitability of water for irrigation. Alkalinity measurements are used in the interpretation and control of water and wastewater treatment processes. Raw domestic wastewater has an alkalinity less than or only slightly greater than that of the water supply.

Requirements:

Apparatus:

Burette, conical flask, pipette, measuring cylinder

Reagents:

H2SO4 solution, Phenolpthalein indicator, Methyl Orang indicator

Procedure:

1. Fill the burette to H2SO4 solution.

2. Take a 100ml water sample in flask. Add few drop of Phenolphthalein indicator.

3. Note the initial reading on burette scale. Titrate against H2SO4 till the pink colour disappear.

4. Note the end point reading and get volume of used H2SO4 in ml(P) (Concordant value I).

5. Add 1-3 drop of Methyl Orange in same sample flask.

6. Titrate it, till the appearance of light orange colour.

7. Note down the final reading and find the volume of used H2SO4.

8. Repeat the steps of using the sample to get concordant value (Concordant value II).

9. Calculate the total alkanity of sample

Observation table:

For Concordant value I: Determination of phenolphthalein end point

S.No.	Water Samples	Initial	Final	Volume of H ₂ SO ₄
		value	Value	rundown(ml) P

For Concordant value II:

Determination of methyl orange end point

S.No.	Water Samples	Initial value	Final Value	Volume of H ₂ SO ₄ rundown(ml) T

Calculations:

Total volume of standard H2SO4 used for the titration:

T = Concordant value I + Concordant value II

Thus, Phenolpthalein alkalinity = P*1000/ml sample

Total alkalinity (mg/lit of CaCO3) = T*1000/ml sample

DISSOLVED OXYGEN

Dissolved oxygen (DO) is a measure of how much oxygen is dissolved in the water - the amount of oxygen available to living aquatic organisms. The amount of dissolved oxygen in a stream or lake can tell us a lot about its water quality.Healthy water should generally have dissolved oxygen concentrations of 6.5-8 mg/L and between about 80-120 %.

Sources:

Oxygen dissolves into water from two sources:

1. The atmosphere and 2. From plants in the water.

*Dissolved oxygen (DO) is one of the most vital indicators of water quality. **High DO levels improve the taste of drinking water, but this can cause corrosion in water pipes**. Alternatively, low dissolved oxygen levels below 5.0 mg/L stress aquatic life and creates hypoxic conditions

*Oxygen enters water through diffusion from air, wind and wave action and plant photosynthesis. Dissolved oxygen levels are

increased by **supplementing wind and wave action, adding plants to water and exposing water to purified oxygen**.

*Solubility of oxygen in water is so small and by diffusion process alone in still water, it was culculated that it would take **6 years** for oxygen to diffuse from surface to a depth of 6 meters in quiet water.

Types of Environment depending on quantity of dissolved oxygen

1. Oligotropic environment:

The environment rich in dissolved oxygen is called oligotropic environment.

2. Eutropic Environment:

The environment which is in the lack in dissolved oxygen is called eutropic environment.

Uses of Dissolved Oxygen:

1.It is essential for life of fish and aquatic animals.Fish absorb the DO directly from the water into their bloodstream through their gills.

2.It is used for the respiration of aquatic plants.

3.Adequate dissolved oxygen is used for good water quality.

Determination of Dissolved Oxygen in Water Samples:

Dissolved oxygen levels can be measured by a basic chemical analysis method (titration method), an electrochemical analysis method (diaphragm electrode method), and a photochemical analysis method (fluorescence method). The diaphragm electrode method is the most widely used method.

Aim:

Determine dissolved oxygen (DO) of given water sample.

Introduction:

Dissolved oxygen (DO) levels in environmental water depend on the physiochemical and biochemical activities in water body and it is an important useful in pollution and waste treatment process control. Two methods are commonly used to determine DO concentration: (1) The iodometric method which is a titration-based method and depends on oxidizing property of DO and (2) The membrane electrode procedure, which works based on the rate of diffusion of molecular oxygen across a membrane. In the Iodometric method, divalent manganese solution is added to the solution, followed by addition of strong alkali in a glass-stopper bottle. DO rapidly oxidize an equivalent amount of the dispersed divalent manganese hydroxide precipitates to hydroxides of higher valence states. In the presence of iodide ions in an acidic solution, the oxidized manganese reverts to the divalent state, with the liberation of iodine equivalent of the original DO content. The iodine is then titrated with a stranded solution of thiosulfate. The titration end point can be detected visually with a starch indicator.

MnSO₄ + 2KOH Mn (OH)₂ + K₂SO₄

 $Mn(OH)_2 + O MnO(OH)_2 \rightarrow$

 $MnO(OH)_2 + 2H_2SO_4 + 2KI \text{ MnSO}_{\ddagger} + K_2SO_4 + 3H_2O + I_2$

Apparatus: Burette, conical flask, pipette, measuring cylinder.

Reagents:

1. Manganese sulfate solution: Dissolve 480 g MnSO4.4H2O, 400 g MnSO4.2H2O or 364 g MnSO4.H2O in distilled water, filter, and dilute to 1L. The MnSO4 solution should not give a color with starch when added to an acidified potassium iodide (KI) solution.

2. Alkali-iodide-azide reagent

3. Sulfuric acid: One mL is equivalent to ~ 3mL alkali-iodide-azide reagent.

4. Starch solution: Dissolve 2 g laboratory-grade soluble starch and 0.2 g salicyclic acid as preservative in 100 mL hot distilled water.

5. Standard sodium thiosulfate titrant: Dissolve 6.205 g Na2S2O3 .5H2O in distiller water and add 1.5 mL 6N NaOH or 0.4 g solid NaOH and dilute to 1000 ml. Standardize with bijodate solution.

6. Standard potassium bi-iodate solution (0.0021M): Dissolve 812.4 mg KH(IO3) in distilled water and dilute to 1000 mL.

7. Standardization: Dissolve e ~ 2 g KI, free from iodate in an Erlenmeyer flask with 100 to 150 mL distilled water; add 1 mL 6N H2SO4 or a few drops of conc. H2SO4 and 20.00 mL standard biodate solution. Dilute to 200 mL and titrate liberated iodine with thiosulfate titrant, adding starch toward end of titration, when a pale straw color is reached. When the solution is of equal, 20.00 mL 0.025M Na2S2O3 should be required. If not, adjust the Na2S2O3 solution to 0.025M.

Procedure:

1. Collect the water sample without bubbling in 200ml glass bottle.

2. Add 2 ml of manganous sulfate (MnSO4.H2O) solution inserting the tip of pipette tip into the sample because the drops of solution can allow inserting the oxygen into the solution.

3. Add 2 ml of the alkali-iodide-azide reagent by above method.

4. Allow reacting the solutions with the oxygen present in the sample. 5. When precipitates are settled down at the bottom add 2 ml of concentrated sulfuric acid by placing the pipette tip very near to sample surface.

6. Mix well to dissolve the precipitates.

7. Take 50 ml of sample from in a flask.

8. Titrate immediately with sodium thiosulfate solution using starch indicator until blue color disappears and note down the burette reading.

9. Determine the burette reading for blank in the same manner.

Calculations:

D.O. in mg/lit = 8*100*N/V*v

Where: V = Volume of sample taken (ml)

v = Volume of used titrant (ml)

N = Normality of titrant 8 is the constant since 1ml of 0.025N Sodium thiosulphate solution is equivalent to 0.2mg oxygen.

Factors affecting Dissolved Oxygen:

- 1. **Pressure:** As pressure increases the concentration of dissolved oxygen is also increases.
- 2. **Temperature:** As temperature increases dissolved oxygen decreases.
- 3. Salinity : Salty water holds less oxygen than fresh water.
- 4. Elevation: At elevated regions the amount of oxygen increases.
- 5. **PH Value:** At high pH value the dissolved oxygen increases.

CHEMICAL OXYGEN DEMAND

Chemical oxygen demand is the amount of oxygen needed to oxidize the organic matter present in water. Chemical oxygen demand testing is used to determine the amount of oxidation that will occur and the amount of organic matter in a water sample. Chemical oxygen demand testing is also used to determine the amount of inorganic chemicals in a sample.

COD increases as the concentration of organic material increases. It also increases if inorganic compounds susceptible to oxidation by the oxidant (typically dichromate) are present. Water with high COD typically contains high levels of decaying plant matter, human waste, or industrial effluent.

Decrease of COD in Waste Water:

One way to reduce COD in water is to use coagulants and flocculants to bind sludge together. Once they are bound into big enough masses that they can be easily filtered out and deposited into a sedimentation tank for removal. This method does however rely on the use of chemicals, and requires high reoccurring cost.

The good COD of surface water normally ranges from 5 to 20 mg l^{-1} .

DETERMINATION OF COD PRESENT IN WASTE WATER

Aim: Determine chemical oxygen demand (COD) of given sewage samples.

Introduction: Chemical oxygen demand (COD) is used to determine the quantity of pollution in water after wastewater treatment. The higher value of chemical oxygen demand indicates the higher organic pollution in the water sample. Only chemically digest able matter can be determined by the COD test. COD determination takes less time than the Biological Oxygen Demand test. COD is recommended where the polluted water has toxicity and organic matter can't be determined by biological oxygen demand and useful in water effluent treatment plants.

The organic matter, present in the water sample is oxidized by potassium dichromate in the presence of sulfuric acid, silver sulfate and mercury sulfate to produce carbon dioxide (CO2) and water (H2O). The quantity of potassium dichromate used is calculated by the difference in volumes of ferrous ammonium sulfate consumed in blank and sample titrations. The quantity of potassium dichromate used in the reaction is equivalent to the oxygen (O2) used to oxidize the organic matter of wastewater.

Requirements:

Apparatus: Burette, conical flask, pipette, measuring cylinder.

Reagents:

1. Potassium dichromate (K2Cr2O7) Solution: Add 6.13 gm Potassium dichromate (previously dried at 105 °C for at least two hours) into 800 ml distilled water. Shake the flask well to dissolve the content and make up the solution to 1000 ml and mix well.

2. Silver sulfate-Sulfuric acid Solution: Dissolve 10 gm Silver sulfate (Ag2SO4) in 500 ml concentrated sulfuric acid and make up the solution to 1000 ml swirl the flask to mix well. Allow standing the solution for 24 hours before use.

3. Mercury sulfate Solution: Dissolve carefully 0.1 gm of HgSO4 in5 ml of concentrated Sulfuric acid.

4. Ferrous ammonium sulfate Solution (0.025 M): Dissolve 9.8 g ferrous ammonium sulfate in a solution of 100 ml of distilled water and 20 ml concentrated Sulfuric acid. Cool the solution and make up the solution to 1000 ml of distilled water. Standardize the solution to determine the actual concentration to calculate the chemical oxygen demand.

5. Ferroin Indicator: 3.5 gm of Iron Sulfate heptahydrate and 7.5 gm of Phenanthroline monohydrate to 400 ml of distilled water. Mix well to dissolve and make up to 500 ml of distilled water.

Procedure:

1. Take 10 ml of sample into a round bottom reflex flask.

2. Add some glass beads to prevent the solution from bumping into the flask while heating.

3. Add 1 ml of Mercury sulfate (HgSO4) solution to the flask and mix by swirling the flask.

4. Add 5 ml of Potassium dichromate (K2Cr2O7) solution.

5. Now add slowly and carefully 15 ml Silver sulfate- Sulfuric acid solution.

6. Connect the reflex condenser and digest the content using a hot plate for 2 hours.

7. After digestion cools the flask and rinses the condenser with 25 ml of distilled water collecting in the same flask.

8. Add 2-4 drops of ferroin indicator to the flask and titrate with ferrous ammonium sulfate solution to the endpoint.

9. Make the blank preparation in the same manner as sample using distilled water instead of the sample.

Observation Table:

S.No.	Sample (ml)	Initial reading	Final Reading	Volume of titrant used(ml)
1.	Water Sample			
2.	Blank			

Calculations:

Calculate the chemical oxygen demand by following formula:

COD = 8*1000*DF*M*(VB - VS) / Volume of sample (in ml)

Where, DF – Dilution Factor (if applicable)

M – Molarity of standardized Ferrous Ammonium Sulfate solution

VB – Volume consumed in titration with blank preparation

VS-Volume consumed in titration with sample preparation

Determination of Chloride by Mohr method

Aim:

Determination the concentration of silver in precipitation method

Principle :

*Mohr's method used to determines the chloride ion concentration of a solution by titration with silver nitrate $AgNO_3$.

*A soluble chromate salt (K_2CrO_4) is added as the indicator. This produces a yellow color solution as the silver nitrate solution is slowly added, a precipitate of silver chloride formed.

 $Ag(aq) + Cl(aq) \rightarrow AgCl(s)$

*This method use chromate as an indicator, chromate forms a precipitant with Ag ions, but this precipitant has a greater solubility than that of AgCl therefore AgCl is formed first and after all Cl ions is consumed .

*The end point of the titration occurs when all the chloride ions are precipitated, then additional silver ions react with the chromate ions of indicator, potassium chromate, to form a red brown precipitant of silver chromate.

 $2Ag(aq) + CrO4(aq) - 2 \rightarrow Ag2CrO_4(s)$

Chemicals and tools :

 $AgNO_3$, K_2CrO_4 , , NaCl, distilled water, burette, pipette, stand, clump, brush, conical flask, spatula, funnel, volumetric flask, washing bottle, beaker, dropper, balance, watch glass.

Experimental work:

1. Wash the burette with distilled water and small amount of (0.1) N of *AgNO*3.

2.Fill the burette with (0.1) N of AgNO3.

3.Take (5) ml of NaCl by pipette and put it in a conical flask.

4. Add (5) drops of K_2CrO_4 indicator to the conical flask and mix well.

5. Titrate with $AgNO_3$ until the appearance of red-brown precipitate.

6.Repeat the titration 3 times and take the average.

Calculations :

Calculate the concentration of Chloride ion in normality by using the law : $(N \times V)AgNO3 = (N \times V)NaCl$ Volume of AgNO3 (V) = (Average value of V1 + V2 + V3 /3)