



LABORATORY MANUAL  
FOR **Environmental Engineering**  
Subject Code: CEP 1403

DEPARTMENT OF CIVIL ENGINEERING  
NATIONAL INSTITUTE OF TECHNOLOGY MIZORAM

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## EXPERIMENT I: Measurement of pH and Conductivity

### Measurement of pH

**AIM:** To ensure calibration of pH meter and measurement of pH of given water sample

#### **PRINCIPLE:**

Measurement of pH is one of the most important and frequently used tests in water chemistry. The pH of a solution is measured as negative logarithm of hydrogen ion concentration (Eq.1).

$$\text{pH} = -\log [\text{H}^+] \quad \text{Eq. (1)}$$

Where  $\{\text{H}^+\}$  is the concentration (or activity) of hydrogen ion (or proton) in moles per liter (M). At a given temperature, the intensity of the acidic or basic character of a solution is indicated by pH or hydrogen ion concentration. pH values from 0 to 7 are diminishing acidic, 7 to 14 increasingly alkaline and 7 is neutral. The basic Brief Theory of electrometric pH measurement is determination of the activity of the hydrogen ions by potentiometric measurement using a standard hydrogen electrode and a reference electrode. Majority of modern pH electrodes are combination pH electrodes rather than a set of two separate electrodes. The combination pH electrode is virtually a tube within a tube arrangement. The inner tube housing the pH indicator electrode (pH sensing membrane, Ag/AgCl reference electrode, and HCl) and the outer one housing the reference electrode (Ag/AgCl) and its salt bridge. The pH sensing component of the indicator electrode is a glass bulb. When a pH probe is immersed in a sample solution, an electrical potential is developed inside and another electrical potential is developed outside, the difference in the potential is measured and is given as the pH of the sample.

**Calibration of pH meter:** To be certain of accurate and reliable measurements, you need to perform pH meter calibration. This is generally done by measuring different buffer solutions with standardized, well-defined values, and then adjusting the pH meter based on any deviations from the buffer's known pH value. Buffers are solutions that resist changes in pH when small amounts of a strong acid or a base are added. Because they maintain a constant pH, buffers are good reference solutions. Calibration should be performed periodically to maintain the pH data quality.

#### **APPARATUS:**

1. pH meter,
2. Glassware (Beaker)

#### **REAGENTS:**

#### **Buffer Solutions:**

- **pH 4:** It is prepared by dissolving buffer tablet of pH 4 in a 100 mL volumetric flask and diluting it up to the mark with deionized water. Alternatively, it may be prepared

by dissolving 1.021 g of potassium hydrogen phthalate ( $\text{KHC}_8\text{H}_4\text{O}_4$ ) in fresh 100 mL deionized water.

- **pH 7:** Dissolve a buffer tablet of pH 7 in deionized water in a 100 mL volumetric flask and make up to the mark. Alternatively, dissolve 0.340 g of potassium hydrogen phosphate ( $\text{KH}_2\text{PO}_4$ ) and 0.355 g disodium hydrogen phosphate ( $\text{Na}_2\text{HPO}_4$ ) in distilled water in a 100 mL volumetric flask and make up the solution to the mark.

## **PROCEDURE:**

### **Calibration of pH meter:**

1. Plug in the meter and let it warm up for five to ten minutes.
2. Put the selector knob to pH mode.
3. Rinse the electrode with de-ionized water and blot dry using a piece of tissue.
4. Allow all of the buffers to reach the same temperature, since pH readings are temperature dependent. If the buffers are not at 25 °C, temperature compensation is recommended. Measure the temperature of the buffers using thermometer and manually adjust the “temperature compensation knob” to this temperature. Where, ATC probe is available the system automatically transmits the temperature of the buffers to the meter.
5. Place the electrode in the solution of pH 7 buffer. It should be ascertained that the glass electrode membrane is completely immersed in the solution. Electrode should not touch the sides or the bottom of the beaker. Allow the pH value to stabilize and adjust 'set buffer knob' in a manner that the reading matches with buffer pH. Remove the electrode from the buffer.
6. Rinse the electrode with de-ionized water and blot dry using a piece of tissue.
7. Place the electrode in the solution of next buffer i.e. pH 4. Follow the step no. 4 until pH value reaches pH 4. Remove the electrode from the buffer. Similarly, other pH standard such as 10.1 can be used, depending on the expected sample pH range. Generally, for groundwater sample pH 7.0 and 10.0 should be used for calibration purpose.
8. Rinse the electrode with de-ionized water and blot dry using a piece of tissue.
9. Now, the instrument is ready to measure pH of unknown sample.

### **pH Measurement of samples**

1. Make sure that the meter is set to the pH mode.
2. Place the electrode in the sample (30-50 mL) to be tested.
3. The pH of the solution appears in the display. Allow the display to stabilize before taking your reading.
4. Note down the pH and temperature reading.
5. Rinse the pH electrode and place it back in the storage solution.

### **PRECAUTIONS:**

1. Make sure to store a pH electrode in its wetting cap containing electrode fill solution (3 M KCl, purchased or prepared by dissolving 22.37 g KCl into 100 mL DI water). Do not store electrode in DI water.
2. Never touch the membrane of the glass electrode with anything else except soft tissue paper since it is fragile and is easily ruined if scratched or bumped.
3. Always bring the standards and the sample to the same temperature before measurement (preferably room temperature).

### **OBSERVATION TABLE:**

<b>Sample No.</b>	<b>pH of the sample</b>	<b>Temperature of the sample</b>

### **ENVIRONMENTAL SIGNIFICANCE OF pH:**

Determination of pH is one of the important objectives in biological treatment of the wastewater. In anaerobic treatment, if the pH goes below 5 due to excess accumulation of acids, the process is severely affected. Shifting of pH beyond 5 to 10 upsets the aerobic treatment of wastewater. In these circumstances, the pH is generally adjusted by addition of suitable acid or alkali to optimize the treatment of the wastewater. pH value or range is of immense importance for any chemical reaction. A chemical shall be highly effective at a particular pH. Chemical coagulation, disinfection, water softening and corrosion control are governed by pH adjustment. Lower value of pH below 4 will produce sour taste and higher value above 8.5 a bitter taste. Higher values of pH hasten the scale formation in water heating apparatus and also reduce the germicidal potential of chlorine. High pH induces the formation of trihalomethanes, which are causing cancer in human beings.

### **RESULT:**

1. The pH of water sample A is \_\_\_\_\_
2. The pH of water sample B is \_\_\_\_\_
3. The pH of water sample C is \_\_\_\_\_

## Measurement of Conductivity

**AIM:** To calibrate conductivity meter and measure conductivity of given water sample

### **PRINCIPLE:**

Conductivity,  $k$ , is a measure of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions; on their total concentration, mobility, valence and on the temperature of measurement. Since the charge on ions in solution facilitates the conductance of electric current, the conductivity of a solution is proportional to its ion concentration. Conductivity cells of different cell constants are chosen mainly to ensure that the actual resistance between the plates, when the cell is immersed in the solution, is within practical measurable range. It is customarily reported in micromhos per centimeter ( $\mu\text{mho/cm}$ ). Freshly distilled water has a conductivity of 0.5-2  $\mu\text{mho/cm}$ .

### **APPARATUS:**

1. Conductivity meter,
2. Glassware (Beaker)

### **REAGENTS:**

- Conductivity water: Pass distilled water through a mixed bed deionizer and discard first 1000 mL of water. Conductivity of this water should be less than 1  $\mu\text{mho/cm}$ .
- Standard potassium chloride solution, KCl, 0.0100M: Dissolve 745.6 mg anhydrous KCl in conductivity water and dilute to 1000 mL at 25°C. This is the standard reference solution, which at 25°C has a conductivity of 1412  $\mu\text{mhos/cm}$ .

### **PROCEDURE:**

#### **Calibration and measurement of sample:**

1. Rinse the electrode thoroughly and then blot and dry.
2. Immerse the electrode in 0.01M KCl solution and the conductivity should be 1413 micro mhos at 25°C Temperature. Adjust the knob to get the accurate value.
3. Now the instrument is calibrated.
4. Remove the cell from KCl solution and wash it thoroughly with distilled water.
5. Immerse the electrode/cell in the unknown sample whose conductivity is to be determined.
6. Note down the instrument reading.

### **OBSERVATION TABLE:**

Sample No.	Conductivity	Temperature

**ENVIRONMENTAL SIGNIFICANCE OF MINERAL ACIDITY:**

Conductivity can tell us a lot about water quality. Drastic changes in conductivity can be used as an indicator of poor water quality from runoff or other sources of pollution.

As salts and other inorganic chemicals dissolve in water, they break down into ions. When ions and dissolved salts are present in the water, they increase the ability to conduct electricity, therefore as salinity increases in water, so does the electrical conductivity (EC). Pure (distilled) water is a good example of a poor conductor because it does not contain any dissolved salts that provide ions to conduct electricity.

Water contains certain elements or chemicals that affect water quality, these include sodium, magnesium, calcium, and chloride. As salinity and temperature affect conductivity, it is also important to measure these parameters; as salinity and temperature increase, conductivity also increases.

When an increase or decrease is detected on a conductivity meter, it can indicate pollutants, affecting the quality of the water. When a sewage leak or agricultural runoff occurs, it increases the EC because of additional ions such as chloride, phosphate, and nitrate, whereas, events such as an oil spill decrease the EC, however, both have a negative impact on water quality.

**RESULT:**

1. The conductivity of water sample A is \_\_\_\_\_
2. The conductivity of water sample B is \_\_\_\_\_
3. The conductivity of water sample C is \_\_\_\_\_





## **EXPERIMENT II: Determination of Acidity of the water sample**

**AIM:** To determine acidity of the given sample.

**PRINCIPLE:** The mineral acids present in the sample which are contributing acidity can be calculated by titrating or neutralizing samples with strong base NaOH to pH 4.3. The CO<sub>2</sub> and bicarbonates present and contribute CO<sub>2</sub> acidity in the sample can be neutralized completely by continuing the titration to pH 8.2.

**INTERFERENCE:** Colour, turbidity, iron, aluminum or manganese and residual chlorine are prime sources of interference. Colour and turbidity can be avoided using potentiometric titrations. Residual chlorine can be removed by adding sodium thiosulphate, iron, aluminum and manganese is prevented by the addition of Na-K titrate.

### **APPARATUS:**

1. Burette
2. Conical flask
3. Pipettes
4. Burette stand
5. Measuring jar

### **REAGENTS:**

1. Standard sodium hydroxide[0.02N]
2. Phenolphthalein indicator
3. Methyl orange indicator
4. Sodium thiosulphate[0.1N]
5. Carbon dioxide free distilled water

### **PROCEDURE:**

10. Take 20ml of the given sample in an conical flask.
11. Total mineral acidity can be calculated by adding 1 drop of 0.1N sodium thiosulphate solution to remove the residual chlorine if present.
12. Add 2 drops of methyl orange the sample turns to pink.
13. Proceed with titration with NaOH solution until the colour changes to yellow.
14. Note down the volume of NaOH added( $V_1$ )
15. Take another conical flask containing 20ml of water sample add 2 or 3 drops of phenolphthalein.
16. Proceed with titration until the sample turns to pink.
17. Note down the total volume of NaOH added ( $V_2$ ).

### **OBSERVATION TABLES FOR MINERAL ACIDITY ( $V_1$ ):**



S.NO	Vol. of sample taken (ml)	Burette reading (ml)		Vol. of NaOH used (ml) (B-A)
		Initial(A)	Final(B)	
1				
2				

**FORMULAE:**

Mineral acidity due to mineral acids mg/lit as CaCO<sub>3</sub> = V<sub>1</sub> × 1000 / volume of sample taken

**OBSERVATION TABLES FOR TOTAL ACIDITY(V<sub>2</sub>):**

S.NO	Vol. of sample taken (ml)	Burette reading (ml)		Vol. of NaOH used (ml) (B-A)
		Initial(A)	Final(B)	
1				
2				

Total acidity due to CO<sub>2</sub> and Mineral acidity (mg/lit as CaCO<sub>3</sub>) = V<sub>2</sub> × 1000 / volume of sample taken

CO<sub>2</sub> acidity (in mg/lit as CaCO<sub>3</sub>) = Total acidity of CaCO<sub>3</sub> - Mineral acidity of CaCO<sub>3</sub>

**ENVIRONMENTAL SIGNIFICANCE OF MINERAL ACIDITY:**

1. Acidity interferes in the treatment of water.
2. It corrodes pipes [zinc coating of G.I pipes gets dissolved].
3. Aquatic life will be affected.
4. P<sup>H</sup> is critical factor for biochemical reaction. The favorable P<sup>H</sup> is 6.8 to 7.5.
5. Waters contain mineral acidity are so unpalatable.

6. Water having acidity more than 50mg/lit cannot used in RCC works.

**APPLICATIONS OF ACIDITY IN EE PRACTICE:**

1. The amount of CO<sub>2</sub> present in an important factor in determining whether removal by aeration or sample neutralization with lime or sodium hydroxide will be chosen as the treatment method.
2. The size of equipment, chemical requirement, storage space and cost of treatment all depend upon amount CO<sub>2</sub> present.
3. CO<sub>2</sub> is an important consideration is estimating chemical requirements for lime and soda ash softening processes.
4. Most industrial wastes containing mineral acidity must be neutralized before they are subjected to biological treatment or direct discharge into water courses or sewers. Quantities of chemical feeders' storage space and costs are determined from the laboratory data of acidity.

**RESULT:**

4. Mineral acidity due to mineral acids as CaCO<sub>3</sub> \_\_\_\_\_ mg/lit
5. Total acidity due to CO<sub>2</sub> as CaCO<sub>3</sub> \_\_\_\_\_ mg/lit
6. Co<sub>2</sub> acidity as CaCO<sub>3</sub> = \_\_\_\_\_ mg/lit

### **EXPERIMENT III: Determination of Alkalinity of the water sample**

**AIM:** To determine the alkalinity of given sample.

**PRINCIPLE:** Alkalinity can be obtained by neutralizing  $\text{OH}^-$ ,  $\text{CO}_3^{2-}$  and  $\text{HCO}_3^-$  with standard  $\text{H}_2\text{SO}_4$ . Titration to pH 8.3 or decolorization of phenolphthalein indicator will show complete neutralization of  $\text{OH}^-$  and 1/2 of  $\text{CO}_3^{2-}$  while to pH 4.4 or sharp change from yellow to pink of methyl orange indicator while indicating total alkalinity. i.e.  $\text{OH}^-$ ,  $\text{CO}_3^{2-}$ ,  $\text{HCO}_3^-$ .

#### **INTERFERENCE FACTORS:**

Interference caused by colour, turbidity or free chlorine have already been discussed in acidity test. In addition, absorption or loss of  $\text{CO}_2$  during or after sample collection can falsify the results.

#### **APPARATUS:**

1. Burette
2. Conical flask
3. Pipettes
4. Measuring jar
5. Burette stand

#### **REAGENTS:**

1. standard sulphuric acid (0.02N)
2. phenolphthalein indicator
3. methyl orange
4.  $\text{CO}_2$  free distilled water
5. sodium thiosulphate (0.1N)

#### **PROCEDURE:**

1. Take 20ml of the given sample in a conical flask.
2. Add one drop of 0.1N sodium thiosulphate solution to remove the free residual chlorine if present.
3. Add 2 drops of Phenolphthalein indicator the sample turns pink.
4. Titrate with 0.02N sulphuric acid till the solution turns to colorless.
5. Note down the volume of  $\text{H}_2\text{SO}_4$  added ( $V_1$ )
6. The titration is continued after adding 2 to 3 drops of Methylorange indicator the sample turns to yellow.
7. Resume titration till the colour of the solution turns to pink.
8. Note down the total volume of  $\text{H}_2\text{SO}_4$  ( $V_2$ )

#### **OBSERVATION TABLE FOR PHENOPHTHALEIN ALKALINITY ( $V_1$ ):**

S.NO	Vol. of sample taken (ml)	Burette reading (ml)		Vol. of H <sub>2</sub> SO <sub>4</sub> (ml) (B-A)
		Initial(A)	Final(B)	
1				
2				

V<sub>1</sub> =

**FORMULAE:**

1. Phenolphthalein alkalinity (P) (mg/lit) as CaCO<sub>3</sub>  
 = V<sub>1</sub> × normality of H<sub>2</sub>SO<sub>4</sub> × 1000 × 50 / volume of sample taken

**OBSERVATION TABLE FOR TOTAL ALKALINITY (V<sub>2</sub>):**

S.NO	Vol. of sample taken (ml)	Burette reading (ml)		Vol. of H <sub>2</sub> SO <sub>4</sub> (ml) (B-A)
		Initial(A)	Final(B)	
1				
2				

V<sub>2</sub> =

**FORMULAE:**

2. Total alkalinity (T) as CaCO<sub>3</sub> mg/lit  
 = V<sub>2</sub> × normality of H<sub>2</sub>SO<sub>4</sub> × 1000 × 50 / volume of sample taken

Relation between P & T	OH <sup>-</sup>	CO <sub>3</sub> <sup>2-</sup>	HCO <sub>3</sub> <sup>-</sup>
<b>P = 0</b>	0	0	T
<b>P &lt; ½ T</b>	0	2P	T - 2P
<b>P = ½ T</b>	0	2P	0
<b>P &gt; ½ T</b>	2P - T	2T - 2P	0
<b>P = T</b>	T	0	0

**P** = Phenolphthalein alkalinity

**T** = Total alkalinity

#### **PUBLIC HEALTH SIGNIFICANCE:**

1. Highly alkaline water is usually unpalatable and consumers tend to seek other supplies.
2. Chemically treated waters sometimes have rather high P<sup>H</sup> values, which have meet with some objection on the part of consumers.
3. Large amount of alkalinity imparts a bitter taste to water.
4. The principal objection of alkaline water is the reactions that can occur between alkalinity and certain cation in water. The resultant precipitate can foul pipes and other appurtenances of water distribution systems.

#### **APPLICATION OF ALKALINITY DATA IN EE PRACTICE:**

1. Chemical Coagulation of Water and Waste Water  
To neutralize acids produced during flocculation the sample should be alkaline as otherwise further flock formation slowly cases.
2. Water Softening  
To find out the quantity of lime and soda ash required for the removal of hardness, alkalinity should be found out.
3. Corrosion Control  
To control the corrosion due to acids, neutral waters are rendered to alkaline.
4. Effluents of Waste

Water containing excess caustic alkalinity is not to be discharged into natural bodies or sewers.

Excess alkalinity in water is harmful for irrigation, which leads to soil damage and reduce crop yields. Water having an alkalinity content of less than 200mg/lit is desirable for domestic consumption.

**RESULT:**

1. Phenolphthalein alkalinity (P) as  $\text{CaCO}_3 = \underline{\hspace{2cm}}$  mg/lit
2. Total alkalinity (T) as  $\text{CaCO}_3 = \underline{\hspace{2cm}}$  mg/lit
3. Concentration of  $\text{OH}^- = \underline{\hspace{2cm}}$  mg/lit
4. Concentration of  $\text{CO}_3^{2-} = \underline{\hspace{2cm}}$  mg/lit
5. Concentration of  $\text{HCO}_3^- = \underline{\hspace{2cm}}$  mg/lit

## EXPERIMENT IV: Determination And Estimation of Total Hardness and Calcium & Magnesium Hardness

### Aim:

To determine the total hardness, calcium, and magnesium hardness of given water sample.

### Apparatus:

Burette with Burette stand and porcelain tile, pipettes with elongated tips, pipette bulb, conical flask (Erlenmeyer Flask) 250 mL, standard flask, wash bottle.

### Reagents:

Ammonia buffer, Eriochrome black T, Inhibitor, 0.02N EDTA, 2N NaOH, murexide indicator

### Principle:

Hardness is determined by the EDTA method in alkaline condition; EDTA and its sodium salts form a soluble chelated complex with certain metal ions. Calcium and Magnesium ions develop wine red colour with Eriochrome black T in aqueous solution at  $\text{pH } 10.0 \pm 0.1$ . When EDTA is added as a titrant, Calcium and Magnesium divalent ions get complexed resulting in sharp change from wine red to blue which indicates endpoint of the titration. Magnesium ion must be present to yield satisfactory point of the titration. Hence, a small amount of complexometrically neutral magnesium salt of EDTA is added to the buffer. The sharpness of the end point increases with increasing pH. However, the specified pH of  $10.0 \pm 0.1$  is a satisfactory compromise. At a higher pH i.e. at about 12.0  $\text{Mg}^{++}$  ions precipitate and only  $\text{Ca}^{++}$  ions remain in solution. At this pH murexide (ammonium purpurate) indicator forms a pink colour with  $\text{Ca}^{++}$ . When EDTA is added  $\text{Ca}^{++}$  gets complexed resulting in a change from pink to purple which indicates end point of the reaction. To minimise the tendency towards  $\text{CaCO}_3$  - precipitation limit the duration of titration period to 5 minutes.

### Procedure:

#### Total hardness

- Take 20mL well mixed water sample in a conical flask.
- Add 1-2mL buffer solution in water sample
- Add a pinch of Eriochrome black T and titrate with standard EDTA (0.02N) till wine red colour changes to blue, note down the volume of EDTA required.
- Calculate volume of EDTA required by sample ( $V_1$ ).



**OBSERVATION TABLE:**

Sample No	Volume of sample (ml)	Burette Reading		Volume of EDTA $V_1$ (ml)
		Initial	Final	

Calculation:

a. Total hardness as  $\text{CaCO}_3$  mg/L =  $V_1 \times 1000 / \text{mL sample}$

Where,  $V_1$  = volume of EDTA required by sample.**Calcium hardness**

- Take 20 ml sample in a porcelain dish.
- Add 1mL NaOH to raise pH to 12.0 and a pinch of Murexide indicator.
- Titrate immediately with EDTA till pink colour changes to purple.
- Note the volume of EDTA required.
- Calculate the volume of EDTA required by sample ( $V_2$ ).

**OBSERVATION TABLE:**

Sample No	Volume of sample (ml)	Burette Reading		Volume of EDTA ( $V_2$ )(ml)
		Initial	Final	

Calculation:

b. Calcium hardness  $\text{CaCO}_3$  as mg/L =  $V_2 \times 1000 / \text{mL sample}$

 $V_2$  = volume of EDTA used by sample.

Total hardness as  $\text{CaCO}_3$ , mg/L = Magnesium hardness as  $\text{CaCO}_3$ , mg/L +  
Calcium hardness as  $\text{CaCO}_3$ , mg/L

c. Magnesium hardness as  $\text{CaCO}_3$ , mg/L = Total hardness as  $\text{CaCO}_3$ , mg/L -  
Calcium hardness as  $\text{CaCO}_3$ , mg/L

**Environmental Significance:**

- Scales are formed as inner coating of the pipelines prevents corrosion.
- Absolutely soft waters are corrosive and dissolve the metals.
- More cases of cardio vascular diseases are reported in soft water areas.
- Hard water is useful to growth of children due to the presence of calcium.
- Hard waters cause excessive consumption of soap used for cleaning purpose.

**Result:** Total hardness in a given water sample is.....mg/Lit as  $\text{CaCO}_3$

Calcium hardness in a given water sample is..... mg/Lit as  $\text{CaCO}_3$

Magnesium hardness in a given water sample is.....mg/Lit as  $\text{CaCO}_3$

## EXPERIMENT V: Determination of Chlorides in a water sample

### Aim:

To determine chloride ion concentration in a water sample.

### Apparatus:

Burette, conical flask, pipette, measuring cylinder.

### Reagents:

- Standard silver nitrate solution (N/50)
- Indicator potassium chromate solution.

### Principle:

Chlorides are present in water usually as NaCl, MgCl<sub>2</sub> and CaCl<sub>2</sub>. Although chlorides are not harmful as such, their concentrations over 250 ppm impart a peculiar taste to the water thus rendering the water unacceptable for drinking purposes. By Mohr's method, chloride ions in a water sample (neutral or slightly alkaline) can be determined by titrating it against standard silver nitrate (AgNO<sub>3</sub>) solution using potassium chromate (K<sub>2</sub>CrO<sub>4</sub>) as an indicator. The pH should be between 7-8. At higher pH, silver ions are precipitated as silver hydroxide. At lower pH, potassium chromate indicator is converted to potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>). Mohr's method is based on the precipitation titration in which silver nitrate solution is released from the burette to the water sample which contains chloride ions and indicator. The silver ions (from silver nitrate solution) react with chloride ions (from water sample) and chromate ions (from indicator) to form white precipitate of silver chloride and red precipitate of silver chromate. The following reactions depict the detection of chlorine:



## INTERFERENCE

Bromide, Iodide, Cyanide, Sulphide, Thiosulphate, Sulphate, Iron, Phosphate are prime sources of interference.

### Procedure:

#### Calibration

Transfer 25 ml of the distilled water in a conical flask and add 3-4 drops of indicator potassium chromate solution. Slowly add standard silver nitrate solution from the burette and shake the solution well. At the end point, light yellow colour starts changing to red colour. The titration is repeated until a concordant volume is obtained. The blank correction for the indicator should be subtracted from the volume of the titrant obtained after titrating them with the unknown water sample solution. Let us consider the titrated volume as (V<sub>1</sub>)

#### Experimental Procedure

- Take 50 ml sample in a conical flask. Measure sample pH (to check for acidity or basicity of the sample, this step is optional).
- Add 1.0 ml of freshly prepared indicator solution of K<sub>2</sub>Cr<sub>2</sub>O<sub>4</sub>.

- Titrate with standard silver nitrate solution until the brick red precipitate appears. This stage marks the end point of titration and note down volume of titrant used. Note the difference of volume as (V<sub>2</sub>). Find the actual volume (V) of titrate used using: V = V<sub>2</sub> - V<sub>1</sub> equation.

**Calculations:**

Calculate chloride ion concentration using the following equation:

$$\text{Chloride Ion Concentration (mg/L)} = (A \times N \times 35.45) \times 1000 / V_{\text{sample}} \text{ Eq. (3)}$$

Where, A = volume of titrant used, N is normality of silver nitrate (here we used N/50 or 0.02 N), and V<sub>sample</sub> is volume of sample used (mL) (here we used 50 ml).

**OBSERVATION TABLE:**

Sl.No	Volume of given water sample in ml	Burette Readings in ml		Volume of AgNO <sub>3</sub> solution in ml
		Initial	Final	

**Environmental Significance:**

Chloride ions (Cl<sup>-</sup>) in water possess multifaceted environmental significance. Naturally occurring from sources like rock weathering and ocean spray, elevated chloride levels often signal pollution from human activities such as road salt runoff, industrial discharge, and agricultural effluents. These heightened concentrations can disrupt freshwater ecosystems by harming aquatic organisms through osmoregulatory stress and metal mobilization in sediments, while also accelerating corrosion of infrastructure, particularly in coastal areas where saltwater intrusion threatens freshwater resources. Moreover, excessive chloride in drinking water raises public health concerns, necessitating robust monitoring to assess water quality and identify pollution sources. Overall, effective management strategies are imperative to mitigate chloride pollution's adverse impacts and safeguard both aquatic ecosystems and human well-being.

**Result:** Total chloride ion concentration in a given water sample is.....mg/Lit

## EXPERIMENT VI: Determination of Residual Chlorine in a water sample

### Aim:

To determine the residual chlorine in a given water sample by iodometric method.

### Apparatus:

Burette with Burette stand and porcelain tile, pipettes, conical flask (Erlenmeyer Flask) 250 mL, standard flask, wash bottle.

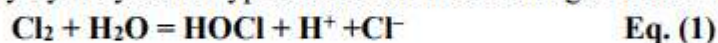
### Reagents:

Bleaching powder solution, Potassium iodide crystal (KI), Glacial acetic acid, Starch indicator, Sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3$ )

### Principle:

The chlorination of water supplies and polluted waters serves primarily to destroy or deactivate disease-producing micro-organisms. A secondary benefit, particularly in treating drinking water, is the overall improvement in water quality resulting from the reaction of chlorine with ammonia, iron, manganese, Sulphide and some organic substances. Chlorine applied to water in its molecular or hypochlorite form initially undergoes hydrolysis to form free chlorine consisting of aqueous molecular chlorine, hypochlorous acid and hypochlorite ion. Disinfectant capabilities of chlorine depend on its chemical form in water, which in turn is dependent on pH, temperature, organic content of water, and other water quality factors. Chlorine is used in the form of free chlorine [e.g., chlorine gas] or as hypochlorites [e.g.,  $\text{NaOCl}$  and  $\text{Ca}(\text{OCl})_2$ ].

Chlorine gas rapidly hydrolyzes to hypochlorous acid according to following reaction:



The two chemical species formed by chlorine in water, hypochlorous acid ( $\text{HOCl}$ ) and hypochlorite ion ( $\text{OCl}^-$ ), are commonly referred to as “free” or “available” chlorine. The difference between the amount of chlorine added to the water and the amount of residual chlorine (i.e., free and combined available chlorine remaining) at the end of a specified contact period is termed as “*chlorine demand*”. Free chlorine (hypochlorite ions and hypochlorous acid) and chloramines stoichiometrically liberate iodine from potassium iodide at pH 4 or less. Residual chlorine is determined by the starch-iodine method as described below:



The starch-iodine method is based on the oxidizing power of free and combined chlorine residuals to convert iodide ion into free iodine at pH 8 or less, as shown below.



The liberated iodine is titrated with a standard solution of sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) with starch as the indicator. The liberated iodine is directly proportional to the concentration of chlorine present in sample. Titrate at pH 3 to 4 because the reaction is not stoichiometric at neutral pH due to partial oxidation of thiosulphate to sulphate. In the starch-iodine method, the quantity of chlorine residuals is determined by measuring the quantity of iodine by titration with a reducing agent sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ). The end point of titration is indicated by the disappearance of blue colour, produced by the reaction between iodine and starch (which is added as indicator during the titration).

**Procedure:**

1. Take 100ml of sample in a conical flask and add a pinch of potassium iodide.
2. Add 5ml of acetic acid and allow the reaction to complete.
3. Titrate the sample against 0.0025N of sodium thiosulphate solution until the yellow colour disappears.
4. Add 1ml of starch solution, blue colour appears then continue the titration until the blue colour disappears (A-B).

**OBSERVATION TABLE:**

SL NO	Sample taken	Burette reading		Difference	Residual chlorine in (mg/l)
		Initial reading	Final reading		
01	Sample 01				
02	Sample 02				

**Calculation:**

Residual chlorine =  $((A-B) \times 0.0025 \times 35.45 \times 1000) / (\text{volume of sample})$ .

**Environmental Significance:**

- Residual chlorine test checks water quality, especially in treated water for drinking.
- It ensures disinfection effectiveness, preventing waterborne diseases and protecting public health.
- Monitoring residual chlorine levels prevents microbial regrowth in the water distribution system.
- Maintaining appropriate chlorine levels minimizes stress on aquatic ecosystems

and protects aquatic organisms.

- Regulatory compliance with residual chlorine testing ensures adherence to drinking water quality standards.
- The test helps optimize disinfection practices while minimizing environmental impacts like disinfection by-product formation.

**Result:** Residual chlorine in the given sample is.....mg/Lit



**EXPERIMENT VII: Determination of Total, Total Suspended, Total Dissolved Solids,  
Total Fixed and Volatile Solids**

**TOTAL, TOTAL SUSPENDED, TOTAL DISSOLVED SOLIDS**

**Aim:**

To determine the total, total suspended, total dissolved solids in given water sample.

**Apparatus:**

Total solids: Evaporating dish, oven, and desiccators.

Total suspended solids: Evaporating dish, whattman filter paper and Hot air oven.

Total dissolved solids: Crucible, filter paper and hot air oven.

**Principle:**

Total solids are determined as a residue left after evaporation and drying of the un- filtered. sample. A well-mixed sample is filtered through filter paper and the residue retained on the filter is dried to a constant weight at 103°C. The increase in weight of filter paper represents the total suspended solids.

**Procedure for total solids:**

- 1) Take 100ml of well mixed sample and pour it into evaporating dishes which is already been heated in an oven at 103<sup>0</sup>C for removing the moisture and desiccated for balancing the temperature and weighed ( $W_1$ ).
- 2) Heat the sample until it is dried(24hrs).
- 3) Take out the evaporating dish ported in a desiccators and take out the final reading ( $W_2$ ).

**OBSERVATION:**

Weight of the empty dish,  $W_1$  = g.

Weight of the sample with dish (oven dried),  $W_2$  = g.

Volume of the sample taken, V = g.

**CALCULATION:**

Initial weight of the evaporating dish ( $W_1$ ) = g.

Final weight of the evaporating dish ( $W_2$ ) = g.

Total solids =  $((W_2 - W_1) \times 1000 \times 1000) / (\text{volume of sample})$ . =mg/l.

**Procedure for total suspended solids:**

- 1) Take a wattman filter paper.
- 2) Place in an oven and heat it at  $103^{\circ}\text{C}$  to remove the moisture. Take a filter paper from the oven placed in desiccators to balance the temperature and take the initial weight ( $W_1$ ).
- 3) Pour known volume of well mixed sample to the filter paper.
- 4) Once after the completion of filtration take the filter paper place it in an the oven and heat it for  $103^{\circ}\text{C}$  for 1 hour.
- 5) Take out the filter paper from oven and place it in the desiccators to balance the temperature and note down the final reading ( $W_2$ ).

**OBSERVATION AND CALCULATION:**

Empty weight of filter paper,  $W_1 =$  \_\_\_\_\_ g.

Weight of filter paper + suspended solids,  $W_2 =$  \_\_\_\_\_ g.

Volume of sample = \_\_\_\_\_ ml.

Total suspended solids =  $((W_2 - W_1) * 1000 * 1000) / (\text{volume of sample})$ .

= \_\_\_\_\_ mg/l.

**Procedure for total dissolved solids:**

Take an evaporating dish, heat it in the oven to remove the moisture, place it in a desiccator to balance the temperature and take the initial weight  $W_1$ . Take known volume of well mixed sample and filter it from a filter paper which is previously dried and weighed evaporating dish. The filtrate left over in an evaporating dish is dried at  $103^{\circ}\text{C}$  and desiccated for balancing the temperature and weight take the final weight  $W_2$ .

**OBSERVATION AND CALCULATIONS:**

Empty weight of evaporating dish,  $W_1 =$  \_\_\_\_\_ g.

Weight of sample with dish after filtration (after oven drying),  $W_2 =$  \_\_\_\_\_ g.

Volume of sample = \_\_\_\_\_ ml.

Total dissolved solids =  $((W_2 - W_1) * 1000 * 1000) / (\text{volume of sample})$ .

= \_\_\_\_\_ mg/l.

**Results:**

Total solids in a given water sample is.....mg/Lit

Total suspended solids in a given water sample is.....mg/Lit

Total dissolved solids in a given water sample is.....mg/Lit

## TOTAL FIXED AND VOLATILE SOLIDS

**Aim:** To determine the Total fixed and volatile solids of the given sample

**Apparatus:**

1. Evaporating dish.
2. Oven 103°C
3. Muffle furnace 600°C
4. Desiccators
6. Water Bath

**Principle:**

Total volatile solids and fixed solids are determined as residue remaining after evaporation, drying at 103°C and ignition at 600°C.

**PROCEDURE:**

1. A clean porcelain dish is ignited in a muffle furnace and after partial cooling in air, it is cooled in a desiccators and weighed ( $W_1$ ).
2. A 100 ml of well mixed sample (graduated cylinder is rinsed to ensure transfer of all suspended matter) is placed in the dish and evaporated at 100°C on water bath, followed by drying in oven at 103°C for 1 hour.
3. Dry to a constant weight at 103°C, cool in desiccator and weighed ( $W_2$ ).
4. Ignite the residue on evaporation at 600°C in the muffle furnace to constant weight in 10 to 15 min.
5. Allow the dish to cool and moisten the ash with a few drops of distilled water.
6. Dry to constant weight at 104°C, cool in a desiccators and weighed ( $W_3$ ).

**CALCULATIONS:**

Total solids (mg/l) =  $((W_2 - W_1) \times 1000 \times 1000) / (\text{volume of sample})$ .

= mg/l.

Total volatile solids (mg/l) = Total solids - Fixed solids.

= mg/l.

Total fixed solids (mg/l) =  $((W_3 - W_2) \times 1000 \times 1000) / (\text{volume of sample})$ .

= \_\_\_\_\_ mg/l.

**OBSERVATIONS:**

Type of solids	Sample details	Volume of sample, ml	Weight of empty dish (mg)	Weight of empty dish + Residue (mg)	Residue (mg/l)

**Result:**

- The amount of Total solids of the given sample is= \_\_\_\_\_ mg/l
- The amount of fixed solids of the given sample is= \_\_\_\_\_ mg/l
- The amount of volatile solids of the given sample is= \_\_\_\_\_ mg/l

## EXPERIMENT VIII: Determination Of Optimum Dosage of Coagulant for a water sample (Jar Test)

**Aim:** To find the optimum dose of coagulant required for treating the given turbid water sample.

**Apparatus:**

1. Jar test Apparatus.
2. pH meter.
3. One-liter beakers - 6 Nos.
4. Graduated pipette.
5. Turbidity meter.

**Reagents:**

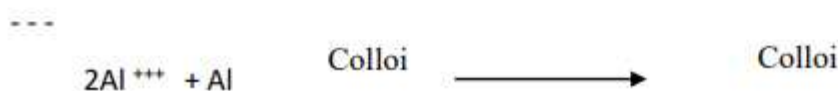
Alum solution- Dissolve 1 g of fresh alum in 1000 ml of distilled water.

**Principle:**

Very fine particles of size 1 to 500 nano meters of clay, micro-organisms, decomposing organic matter, phosphates, fluorides and certain toxicants remain suspended in water without settling and are called **COLLOIDS**.

Colloids:      Hydrophobic (water hating)  
                    Hydrophilic (water loving)

Hydrophobic colloids - Possessing no affinity for water are dependent on electrical charges for their stability in suspension. A coagulant destabilizes these colloids such that they contact agglomerate, form flocs and drop out of solution by sedimentation.



**Flash mixing** helps the coagulant intimately get mixed with colloids and then gentle mixing helps the particles to contact, and then to agglomerate.

**Coagulation** is dependent on pH, colour, turbidity, mineral matter, temperature, time of flocculation and degree of agitation.

The minimum concentration that effectively removes all the turbidity is the ideal dose of the coagulant.

**Procedure:**

1. Measure the turbidity of given sample.



2. Take 1 litre of sample into each of 6 beakers.
3. Switch on the motor and adjust the speed of paddles to 100 rpm.
4. Add varying doses of alum solution i.e., 1 ml, 2ml, 3ml, 4ml, 5ml, 6ml to different beakers simultaneously. (The doses vary with turbidity in water sample).
5. Allow flash mix for 1 minute.
6. Reduce the speed of paddles to 40 rpm and continue mixing for 10 minutes.
7. Switch off the motor and allow 20 minutes for settling of flocs.
8. Collect the supernatant without disturbing the sediment and find the turbidity of each beaker.
9. Repeat the experiment with high doses of alum if satisfactory results are not obtained.
10. Plot a graph of turbidity removed v/s dosage of alum added.
11. Note the ideal (optimum) dose of the coagulant for excellent floc formation.

**OBSERVATION TABLE:**

Raw water turbidity (NTU) =

Raw water pH =

Raw water Alkalinity (mg/l) =

**TABULATION:**

Sl. No	Vol. of sample	Beaker No.	Weight of alum added	Initial turbidity NTU	Final turbidity NTU	turbidity removed
1						
2						
3						
4						
5						
6						

**Environmental Significance:**

- The Jar test helps determine the optimal dosage of coagulants and flocculants in water treatment processes.



- By optimizing chemical dosages, it minimizes the amount of chemicals required, reducing costs and environmental impact.
- Efficient coagulation and flocculation improve sedimentation and filtration, enhancing the removal of suspended solids, organic matter, and contaminants from water.
- Clearer water resulting from effective treatment reduces the need for additional disinfection chemicals, lowering overall chemical usage and environmental footprint.
- Proper treatment reduces the release of pollutants and contaminants into receiving water bodies, preserving aquatic ecosystems.
- Jar testing also aids in identifying the best treatment strategies to address specific water quality challenges, leading to more sustainable and environmentally friendly water treatment practices.

**Result:** Ideal dosage of coagulant (mg/l) =

## **EXPERIMENT IX: Determination of Dissolved Oxygen by Winkler's Method or Modified Azide Method**

**Aim:** To determine the dissolved oxygen present in given water sample.

### **Apparatus:**

BOD bottle (capacity 300ml), sampling device for collection of sample, burette, pipette and measuring jar.

### **Reagents:**

1. Manganese sulphate
2. Alkali iodide Azide reagent
3. Starch indicator
4. Concentrated sulphuric acid
5. Standard sodium Thiosulphate (0.025N)

### **Principle:**

Oxygen present in sample oxidizes the divalent manganese to its higher valency which precipitates as a brown hydrated oxide after addition of NaOH and K. Upon acidification manganese reacts to divalent static acid and liberates iodine from K equivalent to BOD content in the sample. The liberated iodine is titrated against  $\text{Na}_2\text{S}_2\text{O}_3$  (0.025N) using starch as indicator. If oxygen is absent in sample the  $\text{MnSO}_4$  reacts with alkali to form white precipitate  $\text{Mn}(\text{OH})_2$ .

### **Procedure:**

1. Take a clean and dry BOD bottle and collect 300ml of water sample in it.
2. Tap the neck sample of the BOD bottle to expel air bubble if any.
3. Add 2ml of manganese sulphate and 2ml of alkali iodide azide solution to the BOD bottle. The tap water of the pipette should be below the liquid level while adding the above said solution.
4. Re-stopper with care to exclude air bubble.
5. Mix the content properly by repeatedly inverting the bubble 10-15 times.
6. If oxygen is present then the manganese ion gets converted into a brown colour manganese oxide ( $\text{MnO}_3$ ). After taking and allowing sufficient limit for all oxygen to react, the chemical ppt is allowed to settle thereby having a clear liquid at the top portion.
7. A 2ml of conc. sulphuric acid and mix the sample completely by re-stopping the bottle and inverting it.
8. A dark yellow colour solution is obtained which is immediately titrated against sodium this sulphate solution by taking 203ml in a conical flask until the colour changes to pale yellow (straw yellow).
9. Now add starch as an indicator, the colour of solution turns to blue, continue titration till the colour disappears.

**OBSERVATION TABLE:**

Trial no	Volume of sample	Burette reading		Sodium thiosulphate rundown
		Initial reading	Final reading	
01	200			
02	200			
03	200			

$$\text{Dissolved Oxygen (D.O.) as mg/l} = \frac{N_{\text{Na}_2\text{S}_2\text{O}_3} \times V_{\text{Na}_2\text{S}_2\text{O}_3} \times 8000}{\text{Volume of the sample}}$$

$$N_{\text{Na}_2\text{S}_2\text{O}_3} = \text{Normal of } \text{Na}_2\text{S}_2\text{O}_3$$

$$V_{\text{Na}_2\text{S}_2\text{O}_3} = \text{Volume of } \text{Na}_2\text{S}_2\text{O}_3$$

**Environmental Significance:**

- Adequate DO levels are essential for supporting fish, invertebrates, and other aquatic life, as they rely on oxygen for respiration.
- Low DO levels can result from pollution, organic matter decomposition, or high temperatures, leading to hypoxia or even anoxia, which can harm or kill aquatic organisms.
- Monitoring DO levels helps identify areas of water bodies at risk of oxygen depletion, guiding efforts to mitigate pollution and manage nutrient inputs.
- By maintaining healthy DO levels, water quality can be preserved, supporting biodiversity and ecosystem functioning in aquatic environments.
- DO testing is crucial for assessing the impact of human activities, such as industrial discharge or agricultural runoff, on water quality and aquatic ecosystems.
- Effective management of DO levels through pollution control measures and habitat restoration efforts contributes to the conservation of aquatic habitats and the sustainability of aquatic resources.

**Result:**

Dissolved oxygen present in the given tap water sample= \_\_\_\_\_ mg/lit.

Dissolved oxygen present in the given water sample= \_\_\_\_\_ mg/lit.

## EXPERIMENT X: Determination Of Biochemical Oxygen Demand

**Aim:** To determine the biochemical oxygen demand in the given sample of water.

### Apparatus:

- 1) BOD bottle 300 capacity.
- 2) Incubator, to be controlled at  $20\text{C}\pm 10\text{C}$ .
- 3) Burette,
- 4) Pipette and
- 5) Measuring jar.

### Reagents:

- 1) Phosphate buffer.
- 2) Magnesium sulfate.
- 3) Calcium chloride.
- 4) Ferric chloride.
- 5) Sodium Thiosulfate solution 0.025N.
- 6) Additionally, all reagents listed in DO estimation

### Principle:

BOD is defined as the amount of oxygen required by microorganisms while stabilizing biologically decomposable organic matter in a waste under aerobic conditions. The BOD test is widely used to determine

- 1) The pollution load of waste water,
- 2) The degree of pollution in lakes and streams at any time and their self-purification capacity and
- 3) Efficiency of waste water treatment methods.

Since the test is mainly a bio-assay procedure, involving measurement of oxygen consumed by bacteria while stabilizing organic matter under aerobic conditions, it is necessary to provide standard conditions of nutrient supply, pH, absence of microbial growth inhibiting substances and temperature. Because of the low solubility of  $\text{O}_2$  in water, strong wastes are always diluted to ensure that the demand does not increase the available  $\text{O}_2$ . A mixed group of organisms should be present in the sample: if not, the sample has to be seeded artificially. Temperature is controlled at  $20^\circ\text{C}$ . The test is controlled for 5 days as 70 to 80% of the waste is oxidized during this period.

## Procedure:

### PREPARATION OF DILUTION WATER:

- 1) Aerate the required volume of distilled water in a container by bubbling compressed air for about 15 min to attain DO saturation. Try to maintain the temperature near 20°C.
- 2) Add 1ml of phosphate buffer, magnesium sulphate, calcium chloride and ferric chloride solution for each liter of dilution water. Mix well.
- 3) In the case of the wastes which are not expected to have sufficient bacterial pollution, add seed to the dilution water. Generally, 2ml settled sewage is considered sufficient for 100ml of dilution water.

### DILUTION OF SAMPLE

- 1) Neutralize the sample to pH around 7.0 if it is highly alkaline or acidic.
- 2) Take 50ml of the sample and acidify with addition of 10ml 1+1 acetic acid. Add about 1kg KL. Titrate with sodium thiosulphate 0.025N using starch indicator. Calculate the volume of sodium thiosulphate required per ml of the sample and add accordingly to the sample to be tested for BOD.
- 3) Samples having high DO content i.e. DO 9mg/l, due to either algal growth or some other reason, reduce the DO content by aerating the samples.
- 4) Make several dilutions of the pretreated sample so as to obtain about 50% depletion of DO in dilution water but not less than 2mg and the residual O<sub>2</sub> after 5 days of incubation should not be less than 1mg/lit. prepare dilutions as follows
- 5) Siphon out seeded dilution water in a measuring cylinder or volumetric flask half the required volume. Add the required quantity of carefully mixed sample. Dilute to the desired volume by siphoning dilution water and mix well.
- 6) The following dilutions are suggested:

0.1% to 1%	Strong trade waste
1% to 5%	Raw or settled sewage
5% to 25%	Treated effluent
25% to 100%	River water

Keep 1 bottle for determination for the initial DO and incubate 2 bottles at 20°C for 5 days. See that the bottles have a water seal. Prepare a blank in duplicate by siphoning plain dilution water (without seed) to measure the O<sub>2</sub> consumption in dilution water. Fix DO of the bottles kept for immediate DO determination and blank by adding 1ml MnSO<sub>4</sub> followed by 1ml alkali iodide azide solution as described in the estimation of DO. Determine DO in the sample and in the blank on initial day and after 5 days.

**OBSERVATION TABLE:**

**Initial DO**

Sample	Burette reading		Volume(ml)
	Final reading	Initial reading	
Blank			
10% dilution			
20% dilution			

**Five-Day DO**

Sample	Burette reading		Volume(ml)
	Final reading	Initial reading	
Blank			
10% dilution			
20% dilution			

**CALCULATION:**

$$\text{BOD mg/l} = \frac{((D_5 - D_0) - (C_5 - C_0))}{(\text{ml of sample used in decimals})}$$

Where:  $D_5$  and  $D_0$  = DO in sample after 5 days and initial day respectively

$C_5$  and  $C_0$  = DO in blank after 5 days and initial day respectively

**Environmental Significance:**

- High BOD levels indicate the presence of significant organic pollution, which can deplete oxygen levels in water bodies.
- Oxygen depletion resulting from elevated BOD can lead to hypoxia and negatively impact aquatic organisms, potentially causing fish kills and disrupting entire ecosystems.
- Monitoring BOD levels helps assess the health of water bodies and identify sources of pollution, guiding efforts to improve water quality through pollution control measures and wastewater treatment.
- Lowering BOD levels reduces the demand for oxygen in aquatic environments, supporting healthier ecosystems and biodiversity.
- Effective BOD management contributes to the protection and restoration of aquatic habitats, ensuring the sustainability of water resources and the well-being of aquatic organisms.
- By controlling BOD levels, regulatory agencies and industries can mitigate the environmental impacts of organic pollution and promote the long-term health of aquatic ecosystems.

**Result:** BOD of the sample = \_\_\_\_\_ mg/lit.



## EXPERIMENT XI: Determination Of Chemical Oxygen Demand

**Aim:** To determine the chemical oxygen demand of the given sample.

### Apparatus:

- Reflux apparatus consisting of a flat bottom 250 to 500ml capacity flask with ground glass joint and a condenser.
- Burner or hot plate with temperature regulator.

### Reagents:

- 1) Standard potassium dichromate 0.25N.
- 2) Sulphuric acid reagent.
- 3) Standard Ferrous Ammonium Sulphate 0.25N.
- 4) Ferroin indicator
- 5) Mercuric sulphate

### Principle:

COD test determine the O<sub>2</sub> required for chemical oxidation of organic matter with the help of strong chemical oxidant. The test can be employed for the same purpose as the BOD tests taking into account its limitations. The intrinsic limitation of the test lies in its inability to differentiate between the biologically oxidizable and biologically inert material. COD determination has an advantage over BOD determination in that the result can be obtained in about 5 hrs as compared to 5 days required for BOD test. Further, the test is relatively easy, gives reproducible results and is not affected by interferences as the BOD test.

### Procedure:

- 1) Place 0.3g mercuric sulphate in a reflux flask.
- 2) Add 20ml sample or an aliquot of sample diluted to 20ml with distilled water. Mix well.
- 3) Add pumice stone or glass beads followed by 10ml of std. Potassium dichromate.
- 4) Add slowly 30ml of Sulphuric acid containing Ag<sub>2</sub>SO<sub>4</sub> mixing thoroughly. This slow addition along with swirling prevents fatty acid to escape out due to high temperature.
- 5) Mix well, if the colour turns green, either take fresh sample with lesser aliquot or add more dichromate and acid.
- 6) Connect the flask to condenser. Mix the contents before heating, improper mixing will result in bumping and sample may be blown out.
- 7) Reflux for a minimum 20 min. Cool and then wash down the condenser with distilled water.
- 8) Keep it for cool, make the volume to 150ml by adding distilled water and then titrate against ferrous ammonium sulphate using Ferroin indicator. Sharp colours change from blue green to wine red indicates end point or completion of the titration.
- 9) Reflux blank in the same manner using distilled water instead of sample.



**OBSERVATION TABLE:**

Sample	Burette reading		Volume(ml)
	Final reading	Initial reading	
Blank			
Sample 01			
Sample 02			

$$\text{COD mg/l} = ((A-B) \times N \times 8000) / (\text{ml of sample}).$$

Where; A = ml Ferrous Ammonium Sulphate for blank.  
B = ml Ferrous Ammonium Sulphate for sample.  
N = normality of Ferrous Ammonium Sulphate.

**Environmental Significance:**

- COD provides valuable information about the overall level of organic pollutants and wastewater strength, aiding in the assessment of water quality and pollution levels.
- High COD levels indicate increased organic pollution, which can deplete oxygen levels in water bodies, leading to aquatic habitat degradation and potential harm to aquatic life.
- Monitoring COD helps identify sources of pollution and evaluate the effectiveness of wastewater treatment processes in removing organic contaminants before discharge into the environment.
- By understanding COD levels, industries and wastewater treatment plants can implement measures to reduce pollutant loads, such as implementing cleaner production processes or enhancing treatment methods.
- Lowering COD levels in wastewater reduces the demand for oxygen in receiving water bodies, mitigating the risk of hypoxia and supporting healthier aquatic ecosystems.
- Effective COD management contributes to sustainable water resource management and environmental protection, ensuring the long-term health and viability of aquatic ecosystems.

**Result:** COD of the sample =

