

**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 digestible 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 (CO<sub>2</sub>) and water (H<sub>2</sub>O). 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 (O<sub>2</sub>) used to oxidize the organic matter of wastewater.

**Requirements:**

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

**Reagents:**

1. **Potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) 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 (Ag<sub>2</sub>SO<sub>4</sub>) 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 HgSO<sub>4</sub> in 5 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. **Ferroun Indicator:** Add 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 ( $\text{HgSO}_4$ ) solution to the flask and mix by swirling the flask.
4. Add 5 ml of Potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) 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 ferroun 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:**



**Observation table:**

S. No.	Sample (ml)	Initial Value (Burette Scale)	Final Value(Burette Scale)	Volume of Titrant Used (ml)
1	Water Sample	--	--	--
2	Blank	--	--	--

**Calculations:**

Calculate the chemical oxygen demand by following formula:

$$\text{COD} = 8 \times 1000 \times \text{DF} \times \text{M} \times (\text{V}_B - \text{V}_S) / \text{Volume of sample (in ml)}$$

Where, DF – Dilution Factor (if applicable)

M – Molarity of standardized Ferrous Ammonium Sulfate solution

V<sub>B</sub> – Volume consumed in titration with blank preparation

V<sub>S</sub> – Volume consumed in titration with sample preparation