Aim: To determine the amount of Dissolved oxygren in given water sample by winkler's method

Apparatus Required: Burette, Pipette, Conical flask, Cork, Beaker, Volumetric flask, Glass rod, Weighing bottle

Chemicals Required:

1. Manganous Sulphate Solution ($MnSO_4.4H_2O$)
2. Alkali-iodine Oxide
3. Conc. Sulphuric Acid(H_2SO_4)
4. Starch Solution
5. Sodium Thiosulphate Solution (0.025 N)
6. Potassium Dichromate Solution (0.025 N)

Dissolved Oxygen

- Oxygen is one of the most common dissolved gases in the water
- DO is the most important indicator of the health of a water body
- DO is vital for survival of aquatic life in water bodies.
- A higher dissolved oxygen level indicates a better water quality. If dissolved oxygen levels are too low, some fish and other organisms may not be able to survive.
- Oxygen affects a vast number of other water indicators, not only biochemical but also like the odor, clarity and taste.

Dissolved Oxygen

- D.O. range 5-9 mg/L: Good quality water
- Value below 5mg/L: Aquatic organism become stressed
- Value below 2mg/L: hypoxic water cause "Mass Fish Kill"

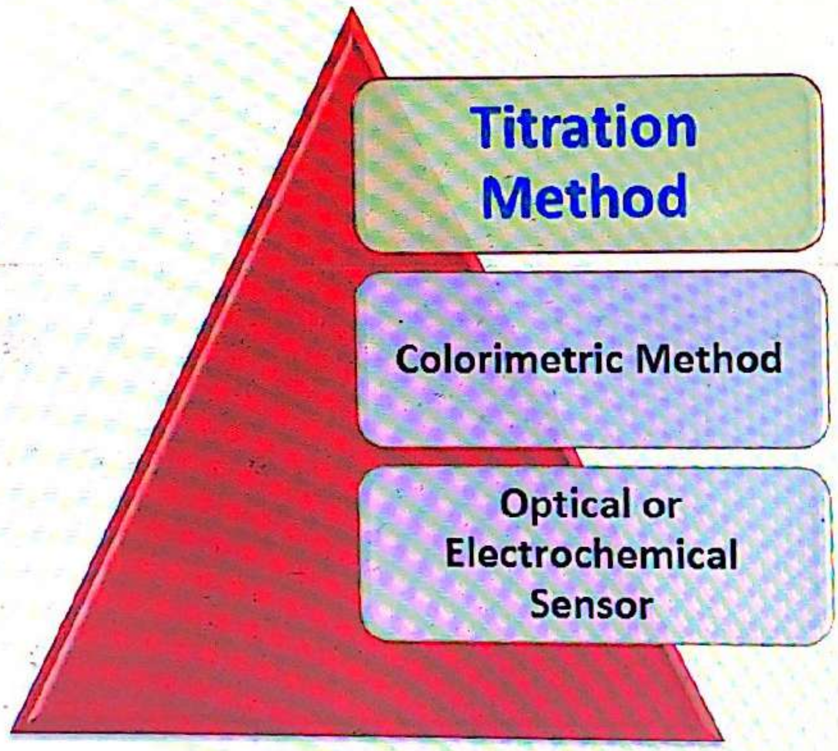
Factor Affecting Dissolved Oxygen

- **Temperature**
- **Pressure**
- **Salinity**
- **Surface area**
- **Level of organic activities**

Oxygen can become dissolved in three ways

- Introduced into water by algae, through photosynthesis
- Enters water directly from atmosphere
- Introduced by mechanical equipment

Dissolved Oxygen Measurement Methods



Titration method/ Winkler method/ Iodometric method for determination of Dissolved Oxygen

- It is not possible to directly measure the amount of dissolved oxygen in a water sample directly.

- The dissolved oxygen does not directly react with another suitable reagent, an indirect procedure was developed by Winkler.

- An iodine/thiosulfate titration can be used to measure the dissolved oxygen present in a water sample.

Titration method/ Winkler method/ Iodometric method for determination of Dissolved Oxygen

1. Preparation of standard solution of $K_2Cr_2O_7$ sample
2. Standardization of $Na_2S_2O_3$
3. Preparation of water sample
4. Titration of water sample against $Na_2S_2O_3$

1. **Preparation of standard solution of $K_2Cr_2O_7$ sample (0.02 N)**
2. **Standardization of $Na_2S_2O_3$:**

Take 10 mL standard $K_2Cr_2O_7$ solution in the conical flask + Conc H_2SO_4 (5 mL) + 5% KI (5 mL).

↓ Keep in dark for 15 minutes

Brown PPt

↓

Titrate with $Na_2S_2O_3$

↓

Pale Yellow Colour

↓ Add 2-3 drops of starch, Titrate with $Na_2S_2O_3$

Colorless

↓

Repeat it till 3-concordant reading

3. Preparation of water sample :

Fill the reagent bottle with water

↓ Remove 5 mL water by pipette+1 mL MnSo4+ 1 mL Alkali Iodide azide

Replace Stopper & invert several times to mix

↓ Flock in the solution is allowed to settle

After settling add 1 mL Conc. H2SO4

↓

Stopped it & invert several times to mix & dissolved all the flock (Repeat it till ppt dissolve)

4. Titration of water sample against $Na_2S_2O_3$:

Take 100 mL water sample

↓

Titrate with $Na_2S_2O_3$

↓

Yellow Colour

↓ Add 2- drops of starch, Titrate with $Na_2S_2O_3$

Colorless solution

↓

Repeat it till 3-concordant reading

Chemical Reactions

Manganese sulfate in alkaline conditions

1. $Mn^{2+}_{(aq)} + 2OH^{-}_{(aq)} \rightarrow Mn(OH)_{2(s)}$ **(white precipitate)**

Mix with sample <u>under water</u> – This reacts with the dissolved oxygen to produce a brown precipitate.

2. $4Mn(OH)_{2(s)} + H_2 0 + O_{2\ (aq)} \rightarrow 4Mn(OH)_{3(s)}$

Adding concentrated H_2SO_4 – enables the Mn(IV) compound to release free iodine from KI.

3. $2Mn(OH)_{3(s)} + 6H^{+}_{(aq)} + 2I^{-}_{(aq)} \rightarrow 2Mn^{2+}_{(aq)} + I_{2(aq)} + 6H_2O_{(l)}$

Titration : The free iodine is then titrated with standard sodium thiosulfate

4. $I_{2(aq)} + 2S_2O_3^{2-}{}_{(aq)} \rightarrow 2I^{-}_{(aq)} + S_4O_6^{2-}{}_{(aq)}$

Table -1 Standardization of $Na_2S_2O_3$ using starch as an indicator

S. No.	Volume of $K_2Cr_2O_7$ (mL)	Burette reading (mL)		Titrant used Difference (mL)
		Initial	Final	
1.	10			
2.	10			
3.	10			
4.	10			
5.	10			

Table -2 Titration of water sample with $Na_2S_2O_3$ using starch as an indicator

S. No.	Volume of Water sample (mL)	Burette reading (mL)		Titrant used Difference (mL)
		Initial	Final	
1.	100			
2.	100			

$MnSO_4$ → Catalyst for the Rx

Alkali azide →

$$N_1 (K_2Cr_2O_7) = \frac{x}{eq\,wt} \times \frac{1000}{100}$$

= —

$$N_1V_1 = N_2V_2$$

$(K_2Cr_2O_7)$ $(Na_2S_2O_3)$

$$N_1 \times 10 = N_2 \times Vol^m \text{ consume}$$

— = N_2

$(N_2V_2) = N_3V_3$

$Na_2S_2O_3$ = water sample

$$N_2 \times Vol^m \text{ consume} = N_3 \times 100 \text{ ml}$$

— = N_3

Amount of Dissolved oxygen = $N_3 \times 1000 \times 8$

= $N_3 \times 1000 \times 8$ = mg/l

N_1= Normality of $K_2Cr_2O_7$

V_1= Volume of $K_2Cr_2O_7$ in conical flask

N_2=Normality of $Na_2S_2O_3$

V_2= Volume of $Na_2S_2O_3$ consumed (from first observation table)

N_3 = Normality of water sample

V_3 = Volume of water sample

V_2 = Volume of $Na_2S_2O_3$ consumed (from second observation table)

RESULT

The Dissolved oxygen of given water sample is found to be ------- mg/L