

**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.



**Requirements:**

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

**Reagents:** 1. Manganese sulfate solution: Dissolve 480 g  $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$ , 400 g  $\text{MnSO}_4 \cdot 2\text{H}_2\text{O}$  or 364 g  $\text{MnSO}_4 \cdot \text{H}_2\text{O}$  in distilled water, filter, and dilute to 1L. The  $\text{MnSO}_4$  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 salicylic acid as preservative in 100 mL hot distilled water.

5. Standard sodium thiosulfate titrant: Dissolve 6.205 g  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  in distilled water and add 1.5 mL 6N NaOH or 0.4 g solid NaOH and dilute to 1000 mL. Standardize with biiodate solution.

6. Standard potassium bi-iodate solution (0.0021M): Dissolve 812.4 mg  $\text{KH}(\text{IO}_3)$  in distilled water and dilute to 1000 mL.

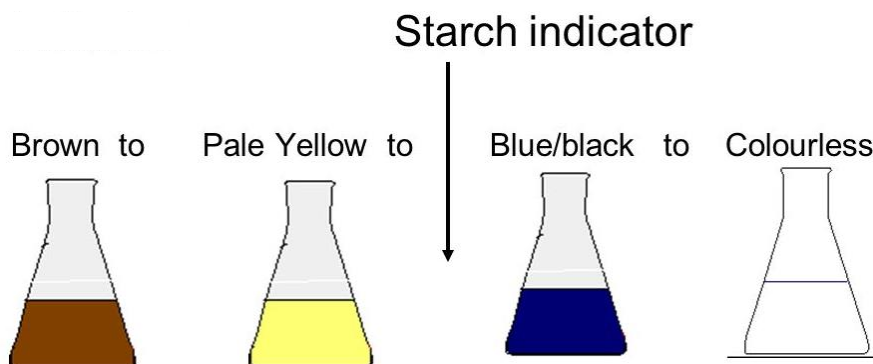
7. Standardization: Dissolve e ~ 2 g KI, free from iodate in an Erlenmeyer flask with 100 to 150 mL distilled water; add 1 mL 6N  $\text{H}_2\text{SO}_4$  or a few drops of conc.  $\text{H}_2\text{SO}_4$  and 20.00 mL standard bi-iodate solution. Dilute to 200 mL and titrate liberated iodine with thiosulfate 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  $\text{Na}_2\text{S}_2\text{O}_3$  should be required. If not, adjust the  $\text{Na}_2\text{S}_2\text{O}_3$  solution to 0.025M.

**Procedure:**

1. Collect the water sample without bubbling in 200ml glass bottle.
2. Add 2 ml of manganous sulfate ( $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ ) 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.

**Observation:**



**Observation table:**

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

**Calculations:**

$$\text{D.O. in mg/lit} = 8 \cdot 100 \cdot \frac{N}{V} \cdot v$$

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.