

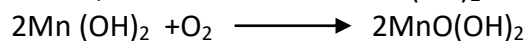
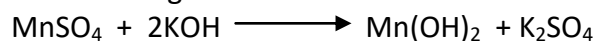
## ESTIMATION OF DISSOLVED OXYGEN IN WATER (WINKLERS METHOD)

### 1.0 AIM

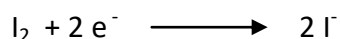
To determine dissolved oxygen content in the given sample of water. You are being provided with sodium thiosulphate solution of approximate strength 0.05N and AR Potassium dichromate crystals.

### 2.0 PRINCIPLE

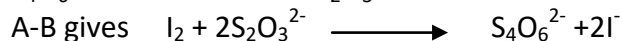
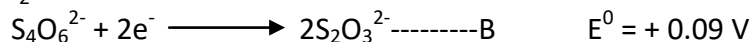
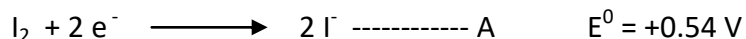
Dissolved oxygen present in water can oxidize  $I^-$  from KI to Iodine ( $I_2$ ). The liberated Iodine can be titrated against  $Na_2S_2O_3$  solution using starch as indicator. Here  $MnSO_4$  in alkaline medium is used to bring about the reaction between KI and dissolved oxygen indirectly.



The above reaction is called *O<sub>2</sub> fixing reaction* and it takes about 15 minutes. After fixing O<sub>2</sub>, the medium is made acidic so that  $Mn(OH)_2$  can oxidize  $I^-$  from KI to Iodine ( $I_2$ ).



The liberated iodine is then titrated with standardised thiosulphate solution.



### 3.0 PROCEDURE

#### 3.1 Preparation of standard Potassium dichromate solution

0.6130 g potassium dichromate crystals are accurately weighed and transferred to a 250ml flask and made up to the mark. The solution will have the normality 0.05 N

#### 3.2 Standardisation of Sodium thiosulphate solution:

20 ml of standard dichromate is pipetted into a conical flask, added about 5ml of 5% KI solution and 3 ml of Con HCl and titrated against sodium thiosulphate solution taken in the burette. When the solution becomes pale yellow, added about 1 ml of freshly prepared starch solution, when the solution becomes blue in colour. The titration is continued by adding sodium thiosulphate solution drop by drop till the blue colour disappears and changes to green.

#### 3.3 Estimation of Dissolved Oxygen in water

Taken a 125 mL of glass bottle (DO Bottle) having air tight stopper. Filled the bottle completely with water without air bubbles. Added 1 mL of aqueous Manganous sulphate ( $MnSO_4$ ) (5 % w/v), and 1 mL of alkaline KI solution into the bottom of the bottle by slowly introducing by a pipette. Closed the lid without having any air bubble and shaken vigorously and allowed to stand for 15 minutes. The precipitate formed during the period is dissolved by adding 1 mL of  $H_2SO_4$  .(1:1) using a pipette.

Closed the lid and shaken the contents of the DO bottle till all the precipitate gets dissolved. The solution is then transferred to a 250 mL conical flask and titrated against standardized sodium thiosulphate solution taken in the burette using starch as indicator as before till the blue colour disappears.

Note the temperature of the water sample collected in the bottle, since the dissolved oxygen content is a function of temperature.

### 4.0 RESULT:

The dissolved oxygen content in the given water sample is ----- PPM

### 5.0 OBSERVATIONS AND CALCULATIONS:

Weight of AR Potassium dichromate crystals. (w) = 0.6130 g

Normality of  $K_2Cr_2O_7$  solution ( $N_1$ ) =  $w/49.04 * 0.25 =$

*Titration 1* :Standardisation of Sodium thiosulphate solution

Standard  $K_2Cr_2O_7$  Vs Sodiumthiosulphate solution

Indicator :starch

S. No	Vol of $K_2Cr_2O_7$ solution $V_1$ (mL)	Burette reading		Vol of sodiumthiosulphate soln. $V_2$ (mL)
		Initial	Final	

Volume of std.  $K_2Cr_2O_7$  solution( $v_1$ ) =                      mL

Normality of std.  $K_2Cr_2O_7$  solution ( $N_1$ ) =

At end point ,  $V_1N_1=V_2N_2$

Normality of Sodium thiosulphate solution( $N_2$ )=  $V_1N_1 / V_2$

*Titration 2* Estimation of dissolved oxygen

Volume of given water sample = 125 mL

Volume of Sodium thiosulphate solution consumed ( $V_3$ ) =

At end point

No of equivalents of dissolved oxygen = No of equivalents of Sodium thiosulphate solution consumed

Normality of water sample( $N$ ) =  $(V_3 * N_2) / 125$

Weight of oxygen per litre of given water sample =  $N * \text{Equivalent mass of oxygen}$   
=  $N * 8 \text{ g}$

Dissolved oxygen content in the given water sample in PPM=  $N * 8 * 1000$   
=                      PPM

Note:

Reagents required for determination of Dissolved Oxygen

1. Alkaline KI is prepared by dissolving 10 g KI in 100 mL of aqueous sodium hydroxide solution(20% w/v).

2. Aqueous Manganous sulphate solution is prepared by dissolving 5 g  $MnSO_4$  in 100 mL water

3  $H_2SO_4$  .(1:1) 100 mL AR Sulphuric acid in 100mL water.

4. Starch solution(5%w/v) : 5 g in 100 mL of water

\*\*\*\*\*