Estimation of Dissolved Oxygen by Winkler's Method

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Principle: Dissolved oxygen is an important factor in corrosion. Oxygen is poorlysoluble in water. The solubility of oxygen decreases with increase in conc. of the salt under a pressure of one atmosphere, the solubility is less in saline water. The estimation of dissolved oxygen in water is useful in studying corrosion effect of boiler feed water and in studying water pollution. *Dissolved oxygen* is usually determined by Winkler's method. It is based on the fact that dissolved oxygen oxidized potassium iodide (KI) to iodine. The liberated iodine is titrated against standard sodium thiosulphate solution using starch indicator. Since dissolved oxygen in water is in molecular state. It as such cannot oxidize KI. Hence Manganese Hydroxide is used as an oxygen carrier to bring about the reaction between KI and Oxygen. Manganese hydroxide, in turn, is obtained by the action of NaOH on MnSO4.

$$MnSO_4 + 2NaOH \longrightarrow Mn(OH)_2 + Na_2SO_4$$

$$2Mn(OH)_2 + O_2 \rightarrow 2MnO(OH)_2$$

$$MnO(OH)_2 + H_2SO_4 \rightarrow MnSO_4 + 2H_2O + [O]$$

$$2KI + H_2SO_4 + [O] \rightarrow K_2SO_4 + H_2O + I_2$$

$$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$$

$$\text{Starch} + I_2 \longrightarrow \text{Blue colored complex.}$$

The liberated iodine (I2) is titrated against standard sodium thiosulphate($Na_2S_2O_3$) solution using starch as indicator.

OBSERVATION AND CALCULATIONS

Titration I :

STANDARDIZATION OF SODIUM THIOSULPHATE

S. No.	Volume of $K_2Cr_2O_7$ (mL)	Burette reading (mL)		Volume of sodium thiosulphate (V_1, mL)
		Initial	Final	
1	20	0	17.6	17.6
2	20	0	17.6	17.6
			Concordant value	17.6

Calculations:

Volume of potassium dichromate V_1 = ~~mL~~ 20mL

Strength of potassium dichromate N_1=~~N~~ 0.01N

Volume of sodium thiosulphate V_2 = 17.6

Strength of sodium thiosulphate N_2= 0.01N

$$V_1N_1 = V_2N_2$$

$$N_2 = V_1N_1/V_2$$

Strength of sodium thiosulphate = N_2 = 0.01N

Titration: II

Estimation of dissolved oxygen

S. No.	Volume of water sample (V1, mL)	Burette reading (mL)		Volume of sodium thiosulphate (V2 ,mL)
		Initial	Final	
1	100	0	8.5	8.5
2	100	0	8.5	8.5
3	100	0	8.5	8.5
			Concordant value	8.5

Calculations :

Volume of sodium thiosulphate V_2 = 8.5 mL

Strength of sodium thiosulphate N_2 = 0.01N

Volume of water sample taken V_1= 100 mL

Strength of given water sample N_1 =

$$V_1N_1 = V_2N_2$$

$$N_1 = V_2 \times N_2/V_1$$

$$= 8.5 \times 10^{-4} N$$

Amount of dissolved oxygen (ppm) = normality × equivalent weight of O_2×1000mg/L

of the given water sample.

$= 8.5 \times 8 \times 1000 \text{ mg/L} \times 10^{-4}$

$= 6.8$ ppm

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Requirements:

Reagents and solutions: Standard buffer of pH 7, standard KCl solution of 0.01 Mconcentration, standard potassium dichromate of 0.01 N, sodium thiosulphate solution, potassium iodide solution, alkali Iodide solution (KI + NaOH in water), conc. H_2SO_4, manganese sulphate, starch solution as indicator,.

Apparatus:, Conical flask, Burette, Measuring flask, Beakers.

TITRATION 1: STANDARDIZATION OF SODIUM THIOSULPHATE

The burette is washed and rinsed with sodium thiosulphate solution. Then the burette is filled with given sodium thiosulphate solution. 20 mL of 0.01N potassium dichromate solution is pipette out into a clean conical flask. To this 5 mL of sulphuric acid and 10 mL of 10% potassium iodide are added. This is titrated against sodium thiosulphate solution, when the solution become straw yellow colour, starch indicator is added and then the titration is continued. The end point is disappearance of bluish brown colour. The titration is further repeated twice or thrice to get the concordant value.

TITRATION 2: ESTIMATION OF DISSOLVED OXYGEN

100 mL of water sample is taken in a conical flask, 2 mL of manganese sulphate and 2mL of alkali iodide solution are added and shaken well for the rough mixing of the reagents. The flask is left aside for few minutes to allow the precipitate to settle down and then 2mL of conc. Sulphuric acid is added for the complete dissolution of the precipitate. Then it is further titrated against standard sodium thiosulphate solution. When the solution becomes light yellow, starch indicator is added. The end point is disappearance of bluish brown colour. The titration is repeated twice or thrice to get the concordant value. From the titre value the strength of dissolved oxygen is calculated and hence the amount of dissolved oxygen in the water sample is calculated.

Results:

Amount of dissolved oxygen in the given water sample = 6.8 ppm