LAB EXPERIMENT – 4

Course: Engineering Chemistry
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Water Quality Monitoring: Total Dissolved Oxygen Assessment in Different Water Samples by Winkler's Method

1. Importance of Dissolved Oxygen (DO): Knowledge of DO concentration in seawater is often necessary in environmental and marine sciences. It is used by oceanographers to study water masses in the ocean. It provides the marine biologist a means to measure primary production, particularly in laboratory cultures. For the marine chemist, it provides a measure of the redox potential of the water column. DO is also an important factor in corrosion. Oxygen is poorly soluble in water. The solubility of oxygen decreases with increase in concentration of the salt and hence, solubility of DO is lesser in saline water. The amount of DO at 100% saturation at sea level is 9.03 mg/L (at 20° C) and is sufficient to sustain aquatic life. Dissolved oxygen is usually determined by Winkler's method.

2. What is Winkler Method? The Winkler Method is a technique used to measure dissolved oxygen in freshwater systems. DO is used as an indicator of the water body's health, where higher DO concentrations are correlated with high productivity and little pollution. This test is performed on-site, as delays between sample collections and testing may result in an alteration in oxygen content.

3. How does the Winkler Method Work? Winkler Method uses titration to determine dissolved oxygen in the water sample. A sample bottle is filled completely with water (no air is left to skew the results). DO in the sample is then "fixed" by adding a series of reagents that form an acid compound that is then titrated with a neutralizing compound that results in a colour change. The point of colour change is called the "endpoint," which coincides with the dissolved oxygen concentration in the sample. DO analysis is best done in the field, as the sample will be less altered by atmospheric equilibration.

4. Applications:

Dissolved oxygen analysis can be used to determine the health or cleanliness of a lake or stream, amount and type of biomass a freshwater, the amount of DO that a system can support and the amount of decomposition occurring in the lake or stream.

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Experiment	**Water Quality Monitoring: Total Dissolved Oxygen Assessment in Different Water Samples by Winkler's Method**
Problem definition	Dissolved oxygen (DO) is essential to living organisms in water but harmful if present in boiler feed water leading to boiler corrosion.
Methodology	DO in water can be assessed using Winkler's titration method.
Solution	Estimation of total dissolved oxygen in different water samples.
Student learning outcomes	Students will learn to a) perform Winkler's titration method b) assess the total dissolved oxygen in different water samples

Principle: Estimation of dissolved oxygen (DO) in water is useful in studying corrosion effect of boiler feed water and in studying water pollution. DO is usually determined by Winkler's titration method. It is based on the fact that DO oxidize potassium iodide (KI) to iodine. The liberated iodine is titrated against standard sodium thiosulphate solution using starch indicator. Since DO in water is in molecular state, as such it 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 $MnSO_4$.

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

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

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

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

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

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

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

Requirements:

Reagents and solutions: Standard buffer of pH 7, standard KCl solution (0.01 M), standard potassium dichromate(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.

Procedure:

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 $K_2Cr_2O_7$solution is pipette out into a clean conical flask. To this, 5 mL $H_2SO_4$and 10 mL of 10% KI 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 $MnSO_4$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. $H_2SO_4$is added for the complete dissolution of the precipitate. It is further titrated against standard sodium thiosulphate solution. When the solution turn light yellow, starch indicator is added. The end point is the disappearance of bluish brown colour. The titration is repeated twice or thrice to get the concordant value. From the titer value, the strength of dissolved oxygen is calculated. Based on that, the amount of dissolved oxygen in the water sample is calculated.

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 (mL)
		Initial	Final	
1	20	0	16.1	16.1
2	20	0	16.2	16.2
3	20	0	16.2	16.2
Concordant value				16.2

Calculations:

Volume of potassium dichromate $V_1 = 20$ mL

Strength of potassium dichromate $N_1 = 0.01$ N

Volume of sodium thiosulphate V_2 =....16.2.....mL (From Titration – 1)

Strength of sodium thiosulphate N_2 = ...?

$$V_1N_1 = V_2N_2$$

$$\therefore\ N_2 = V_1N_1/V_2$$

Strength of sodium thiosulphate = $N_2 = 20 \times 0.01/V_2$ =...0.012.....N

Titration – II: Estimation of dissolved oxygen

S. No.	Volume of water sample (mL)	Burette reading (mL)		Volume of sodium thiosulphate (mL)
		Initial	Final	
1	100	0	5	5
2	100	0	4·7	4·7
3	100	0	4·7	4·7
			Concordant value	4·7

Calculation:

Volume of sodium thiosulphate V_2 = 4·7 mL

Strength of sodium thiosulphate N_2 = 0·012 N (From Titration – 1 calculation)

Volume of water sample taken V_1= 100 mL

Strength of given water sample N_1= ?

$$V_1N_1 = V_2N_2$$

$$N_1 = V_2 \text{ X } N_2/100$$

$$= 5{\cdot}64 \times 10^{-4} \text{ N}$$

Amount of dissolved oxygen (ppm) = normality × equivalent weight ofO_2×1000mg/Lof the given water sample.

$= \cancel{4{\cdot}7}\ 5{\cdot}64 \times 10^{-4}$ N × 8 × 1000 mg/L

= ~~3·76~~ 4·512 ppm.

Result: Amount of dissolved oxygen in the given water sample= 4.512 ppm.

Evaluation of Result:

Sample number	Experimental value	Actual Value	Percentage of error	Marks awarded
	4·512 ppm			