EXPERIMENT 23 ESTIMATION OF DISSOLVED OXYGEN CONTENT OF WATER SAMPLES

Structure

23.1 INTRODUCTION

Oxygen is necessary for aerobic respiration. Aquatic organisms for respiration obtain the oxygen from water, where it remains in dissolved form. In addition the dissolved oxygen in water affects the oxidation-reduction state of many other chemical variables, such as nitrate and ammonia, sulphate and sulphite, and ferrous and ferric ions. The amount of oxygen present in aquatic environment is highly variable and generally low. Many factors such as temperature, salinity, respiration, photosynthesis and decomposition of decaying plants and animals affect the amount of dissolved oxygen. As such oxygen is not very soluble in water and the solubility decreases with increasing temperature. The photosynthetic acitivity of water plants increase the amount of dissolved oxygen during day time, whereas during night it becomes depleted due to respiration of plants and animals. During the process of decomposition microorganisms use the dissolved oxygen thus making it deficient. This adversely affects the other aquatic organisms. You can see in Table 23.1 the oxygen content in some respiratory media.

Table 23.1: Oxygen content of some samples of water and air

Samples	Dissolved Oxygen content millilitres/litre
Sea water at 5° C	6.4
Fresh water at 5° C	9.0
Fresh water at 25° C	5.8
Air	209.5

The amount of oxygen dissolved in water can be measured and is usually expressed as mg/1 (equivalent to parts per million or ppm). There are two methods of estimating dissolved oxygen: by using oxygen electrodes and by Winkler's titration method.

Winkler's method is the most commonly used method for estimation of dissolved oxygen in water. In this lab exercise you will be estimating the dissolved oxygen by Winkler's method from at least from two different water sources such as a pond and a well, or tap water and well water. or a river and pond.

Objectives

At the end of this lab exercise you should be able to:

- describe the principle behind the estimation of the dissolved oxygen in water,
- perform the experimental procedure without any difficulty,
- become familiar with the calculations for the estimation of oxygen, and
- discuss that the oxygen content of the different aquatic habitats differ significantly.

23.2 PRINCIPLE

Winkler's method is a volumetric procedure in which manganous ions (Mn^{2+}) are oxidised into manganic ions (Mn^{3+}) which reacting with an alkali precipitates into $MnO(OH)_2$ and $Mn(OH)_2$. The extent of oxidation is directly related to the amount of dissolved oxygen. In the presence of iodide ions in dilute sulphuric acid, the manganese hydroxide is converted into manganous sulphate [$MnSO_4$] and simultaneously the iodide ions are oxidised to molecular iodine (I_2). It is the concentration of this iodine that is directly proportional to the concentration of oxygen in the original water sample. The amount of iodine liberated at the end of the reaction can be determined by titration with a thiosulphate solution using starch as an indicator to determine the end product.

23.3 MATERIALS REQUIRED

1. Burette and Burette stand
2. 300 ml. glass stoppered reagent bottles
3. 250 ml. conical flasks
4. 10 ml. pipettes
5. Measuring cylinder
6. $MnSO_4$ solution (36 gms of $MNSO_4$ dissolved in 100 ml. of distilled water.
7. Alkaline-iodide solution
   a) 100 gms of NaOH/100 ml. of distilled water
   b) 27 gms of NaI/100 ml. of distilled water
   c) Mix solutions a and b

8. Concentrated H_2SO_4

9. Starch solution 1 gm of slarch per 100 ml. of distilled water. The water musl be heated to bearable warmth and the starch dissolved in it.

10. 0.025N sodium thiosulphate ($Na_2S_2O_3$) solution. (6.205 gms of $Na_2S_2O_3$. $5H_2O$ per 1000 ml. of distilled water).

23.4 PROCEDURE

From each sample obtain water carefully and without air bubbles in 300 ml glass stoppered reagenl bottles. Label the bottles as A and B. For accurate determination of dissolved oxygen it is very necessary that special care in sampling and preparation of water samples should be taken. Any exposure of the sample to air will vitiate your results. Therefore, it is suggested that you collect water by keeping your bottle under the surface of water and allow the water to flow into the bottle very slowly without mixing with the air. It is also necessary that prior to the filling of the sample into the bottle, you determine the volume of the bottle. You may use a measuring cylinder for this purpose. Immediately after collecting the sample close the bottle with a glass stopper. This helps you to eliminate the air spaces. Now, you may add the various reagents to the sample as detailed below:

1. Remove the stoppers and add 2 ml. of $MnSO_4$ solution followed by 2 ml of alkaline-iodide solution in bottles A and B. Addition of these reagents should be done below the surface of water by dipping the pipette into the water thus preventing the contamination with air.

2. Stopper the bottles and gently tilt them several times for the solutions to mix. You will see the formation of yellowish brown precipitates of $Mn(OH)_2$ and $MnO(OH)_2$. Allow the precipitate to settle down and gently shake again.

3. Remove the stopper and add carefully 2 ml of conc. H_2SO_4 under the surface of prepared samples. Stopper the bottles again and mix well. The brown precipitate completely dissolves leaving a straw or brown coloured solution.

4. Transfer 50 ml of the contents of the sample bottle A to a 250 ml conical flask. Add 1 ml of starch indicator solution. The solution turns blue. Titrate this solution against 0.025N sodium thiosulphate solution.

   For titration you have to fill the burette with the thiosulphate solution. Open the stopcock of the burette and let the solution run down once. Refill the burette upto zero mark and perform the titration. The end point is the disappearance of the blue colour. Record the burette reading. You may repeat the titration till you get the concordant values. The concordant values may be obtained even at the end of the second titration if you do them carefully.

5. Repeat the above procedure with the sample B. Fill in the data in your observation note book in the form of the table provided below.

Sample	S. No.	Volume of the sample (ml)	Burette reading initial	final	Volume of $Na_2S_2O_3$ consumed
A	1.	50	0	4.5	4.5
	2.	50	4.5	9.0	4.5

23.5 CALCULATIONS AND RESULTS

You can obtain the amount of dissolved oxygen per litre of water using the following calculations.

$$\text{Amount of oxygen/litre} = \frac{K \times 200 \times \text{vol. of } Na_2S_2O_3 \times 0.698}{\text{Volume of the sample}}$$

$$\text{where } K = \frac{\text{Volume of bottle}}{\text{volume of the bottle} - \text{volume of the reagent added}}$$

A sample calculation is shown below:

Volume of the bottle = 300 ml

Amount of reagent used = 4 ml (2 ml $MnSO_4$ + 2 ml Alkaline iodide)

$$K = \frac{300}{300-4} = \frac{300}{296} = 1.014$$

Volume of NaS_2O_3 consumed = 4.5 ml

$$\text{Amount of } O_2 = \frac{K \times 200 \times 4.5 \times 0.698}{50}$$

$$= \frac{1.014 \times 200 \times 4.5 \times 0.698}{50} = 12.74 \text{ mg/L}$$

23.7 SAQ

Do you **find** any difference in the oxygen content of the two water samples? If the answer is yes, how do you account for the difference?

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