EXPERIMENT NO. 4

Date :

DISSOLVED OXYGEN

AIM OF THE EXPERIMENT : To determine the amount of dissolved oxygen present in a given water sample by Winkler's method.

APPARATUS REQUIRED :

1. Burette 2. Pipette 3. Conical flask 4. B.O.D. bottle

CHEMICALS REQUIRED :

1. Manganous Sulphate Solution : (Winkler's - A)
2. Alkali - Potassium Iodide Solution : (Winkler's - B)
3. Standard $Na_2S_2O_3$ solution (0.01 N)
4. Starch Indicator

PRINCIPLE :

Dissolved oxygen (DO) is determined by Winkler's method or iodometric titration. The dissolved oxygen in water oxidises KI and an equivalent amount of iodine is liberted. The iodine is titrated against a standard hypo solution. However, since dissolved oxygen in water is in molecular state and is not capable of reacting with KI, therefore an oxygen carrier such as manganese hydroxide is used.

The method involves introducing a concentrated solution of $MnSO_4$, NaOH and potassium iodide into water sample. The white precipitate of $Mn(OH)_2$ which is formed, is oxidised by oxygen in water sample to give a brown precipitate of basic manganic oxide $MnO(OH)_2$. The $MnO(OH)_2$ in acidic medium dissolves and liberates free iodine from the added KI in an equivalent amount of dissolved oxygen present in the water sample.

This liberated I_2 is then titrated against $Na_2S_2O_3$ $\left(\frac{N}{100}\right)$ solution using starch as indicator.

CHEMICAL REACTION :

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

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

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

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

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

(Sodium tetra thionate)

IONIC REACTION :

$Mn^{2+} + 2\ OH^- \rightarrow Mn(OH)_2$ (Under zero D.O condition)

$Mn^{2+} + 2\ OH^- + 1/2\ O_2 \rightarrow MnO_2 + H_2O$ (In presence of D.O in the sample)

$MnO_2 + 2\ I^- + 4\ H^+ \rightarrow Mn^{2+} + 2\ H_2O + I_2$

$2\ S_2O_3^{-2} + I_2 \rightarrow S_4O_6^{-2} + 2\ I^-$

PROCEDURE :

1. Collect the supplied water sample in a BOD bottle avoiding as far as possible contact with air.
2. Add 2 ml. of $MnSO_4$ solution (Winkler's-A reagent) followed by 2 ml of alkaline KI (Winkler's-B reagent) with two different pipettes in such a way that the tip of pipette should dip below the water surface.
3. Close the bottle with the stopper immediately and mix the solution well by shaking.
4. Allow the precipitate to settle down.
5. Then add 40-45 drops of conc. H_2SO_4. Close the bottle and shake till all the precipitate dissolved. Allow the solution to stand for 5 minutes.
6. Pipette out 50 ml solution from the B.O.D. bottle and titrate with 0.01 N $Na_2S_2O_3$ solution using starch as indicator till the blue colour is discharged.
7. Repeat this titration to get the concordant reading.

CALCULATION :

Let N_1= Normality of $Na_2S_2O_3$ solution.

V_1= Volume of $Na_2S_2O_3$ solution.

N_2= Normality of water sample due to dissolved O_2.

V_2= Volume of water sample = 50 ml

$$N_2 = \frac{N_1V_1}{V_2}$$

So, Strength of D.O. = N_2 x 8 = ... g/ liter

or $N_2 \times 8 \times 1000 =$..... mg/ liter or ppm.

Questions for Discussion.

1. What is DO and how does it help the users ?
2. What is the necessity of measuring the DO of water sample ?
3. What is the difference between DO and BOD ?
4. What is the difference between Iodometry and Iodimetry ?
5. Which substance acts as an oxygen carrier in this experiment ?
6. Write the reactions involved in this experiment.
7. Amount of dissolved oxygen in a water sample depends on which factors ?
8. Why $MnSO_4$ is added in the experiment ?
9. Why starch is added towards the end point of the titration and not in the beginning ?
10. Why should the bottle be kept in dark for liberation of iodine ?

Rough Work

DEPARTMENT OF CHEMISTRY

Roll No. :

Branch :

Date :

AIM OF THE EXPERIMENT :

OBSERVATION TABLE :

No of obs	Volume of water sample with reagent in ml.	Burette Readings (ml)			Remarks
		Initial	Final	Difference	

CALCULATION :

CONCLUSION :