EXPERIMENT NO: 07

WATER ANALYSIS

AIM: Determination of dissolved Oxygen present in given water sample (Winkler's method.)

THEORY: The principle involved in the determination of D.O. is to bring about the oxidation of potassium Iodide to Iodine with the dissolved oxygen present in the water sample after adding $MnSO_4$, KOH and KI, the basic manganic oxide formed acts as an oxygen carrier to enable the dissolved oxygen in the molecular form to take part in the reaction. The liberated Iodine is titrated against standard sodium thiosulphate (HYPO) solution, using starch as indicator.

$MnSO_4 + 2KOH \rightarrow Mn(OH)_2 + K_2SO_4$

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

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

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

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

Starch + I_2 $\rightarrow$ Blue coloured complex.

In the modified winkler's method, the interference due to certain oxidizing agents such as NO_2^- or reducing agents such as Fe^{+2} or SO_3^{2-} is removed by treating the sample with an excess of $KMnO_4$ in acid medium the following reaction takes place:

$5NO_2^- + 2MnO_4^- + 6H^+ \rightarrow 5NO_3^- + 2Mn^{2+} + 3H_2O$ ----------(1)

$5SO_3^{2-} + 2MnO_4^- + 6H^+ \rightarrow 5SO_4^{2-} + 2Mn^{2+} + 3H_2O$ ----------(2)

$5Fe^{2+} + 2MnO_4^- + 8H^+ \rightarrow 5\,Fe^{+3} + Mn^{2+} + 4H_2O$ ----------(3)

NO_3^- and SO_4^{2-} formed in reaction (1) and (2) do not interfere. Fe^{+3} interferes only when present in amounts above 10mg/lit. Excess of $KMnO_4$ is destroyed by adding potassium oxalate.

$2MnO_4^- + 16H^+ + 5C_2O_4^{2-} \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O$

REQUIREMENTS: Burette, Pipette, Standard sodium thiosulphate (N/50), $KMnO_4$ (N/10),Potassium Oxalate (2%), Manganous Sulphate solution (4.8%), Alkaline potassium Iodide, freshly prepared starch, Conc. H_2SO_4, water sample.

PROCEDURE: Collect water sample in 300ml glass stoppered bottle and with the help of a graduated pipette, add 0.9ml Conc. H_2SO_4 and 0.2ml (4drops) $KMnO_4$ solution. Stopper the bottle and mix the contents of the bottle by inverting it a few times. If the permanganate colour disappears within five minutes, add additional amount of $KMnO_4$. Add 0.5ml of Potassium Oxalate solution, stopper and mix well. Add additional amount of oxalate solution if the permanganate colour is not discharged within ten minutes. Add 2ml of $MnSO_4$ solution followed by 3ml of Alkaline KI solution. Stopper and shake and allow the precipitate to settle. Now add 1ml of conc. H_2SO_4 solution and mix until the precipitate is completely dissolved. Measure 102.2ml of this solution with a measuring cylinder into a conical flask and titrate slowly against N/50 Hypo solution. When the colour of the solution is light yellowish, add about 2ml of freshly prepared starch solution and continue the titration to the disappearance of the blue colour and note down the volume of the Hypo used.

OBSERVATION:

Total volume of the sample taken = 300ml.

Volume of reagents added during = 0.9ml H_2SO_4 + 0.2ml $KMnO_4$ + 0.5ml $K_2C_2O_4$ + 2ml $MnSO_4$ + 3ml alkaline KI = 6.6ml
the preparation of Iodine solution

Volume of prepared solution (Iodine) taken for titration = 102.2ml.

Concordant volume of N/50 Hypo solution used. = Vml

Indicator = Starch solution.

Colour change = Blue to colourless

Burette reading

Reading	1st in ml.	2nd in ml.	3rd in ml.	Mean in ml.
Final	3.2	3.2	3.2	
Initial	0	0	0	
Difference				

CALCULATIONS:

6.6ml of the reagents have been added under such conditions that approximately equal volume of the sample is displaced. This dilutes the sample so a correction is needed.

$N_1V_1 = N_2V_2$

(Oxygen Solution) (Hypo Solution)

$N_1 \times 100 = 1/50 \times V$

or

N_1 (Normality of the sample with respect to D.O.) $= \frac{1}{100} \times \frac{1}{50} V$

$= \frac{1}{100} \times \frac{1}{50} V \times 8 g/lit.$

$= \frac{1}{100} \times \frac{1}{50} \times V \times 8 \times 1000 mg/lit.$

$= 1.6\ V$ mg/lit.

RESULT: The Amount of dissolved Oxygen present in water = _____ ppm.