Experiment- 3

Aim: To determine the total residual chlorine in given water sample.

Apparatus required: Pipette, burette, funnel, conical flask, volumetric flask and beakers etc.

Chemical required: Water sample, N/100 hypo ($Na_2S_2O_3$) solution, N/100 $K_2Cr_2O_7$ (Potassium Dichromate) solution, iodine (KI), H_2SO_4, glacial acetic acid.

Indicator used: starch

Theory: Chlorine is powerful oxidising agent and is cheaply available. The estimation of residual chlorine is based on oxidation of KI by residual chlorine i.e. by iodometric titration.

When the water sample containing residual chlorine (both free and combined) oxidizes KI and liberated I_2 In equivalent amount. Chlorination of water causes the formation of Hypochlorous acid that kills the microbes and other pathogens and rest of it causes residual chlorine as follows:

1. $Cl_2 + H_2 \longrightarrow HOCl$

   $HOCl \rightleftharpoons H^+ + OCl^-$

Here, Cl_2, HOCl & OCl- all account for free chlorine.

2. Water also have NH_3,

   $NH_3 + HOCl \longrightarrow NH_2Cl + H_2O$

   $NH_2Cl + HOCl \longrightarrow NHCl_2 + H_2O$

   $NHCl_2 + HOCl \longrightarrow NCl_3 + H_2O$

All NH_2Cl, $NHCl_2$ & NCl_3 accounts for combined chlorine

This case we are using Redox titration -

It is Iodometric titration which is an in-situ generation of iodine and this liberated iodine is then titrated with hypo solution.

i.e. $Cl_2 + KI \longrightarrow KCl + I_2$

$I_2 + Na_2S_2O_7 \longrightarrow NaI + Na_2S_4O_6$

(Hypo sol^n)

Where, starch is used as indicator (generates blue –black colour with iodine)

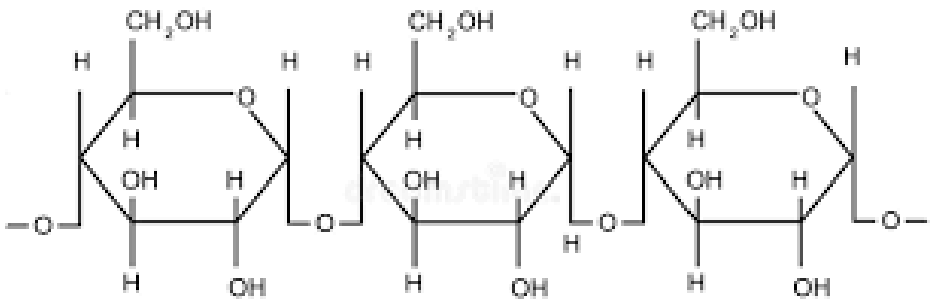
Procedure:

I) **Standardisation of hypo solution: - (Hypo / $K_2Cr_2O_7$)**

1. Pipette out 10ml of $K_2Cr_2O_7$ solution in conical flask add 5ml of H_2SO_4, then 1 spatula KI .Shake the conical flask.
2. Cover it and keep in dark for 10 minutes.
3. Add a pinch of $NaHCO_3$.
4. Titrate it with hypo solution, till the colour changes to pale yellow then add2-3 drop of starch (blue)
5. Again titrate this blue solution with hypo, to obtain light green colour/ colourless. This is the end point.
6. Take 3 concordant reading & calculate the normality of hypo solution.

ii) Titration of hypo with water sample - (**Hypo / Water)**

1. Pipette out 10ml of water sample in conical flask and add 3ml of glacial acetic acid and add one spatula of KI.
2. Cover it and keep it in dark for 10 minutes.
3. Titrate with hypo till pale yellow colour.
4. Add 1-2 drops of starch (blue) to this pale yellow solution to obtain dark blue colour.
5. Again titrate with hypo to obtain colourless solution.



Chemical Structure of Starch (Amylose)

Observation Table:

i)Standardisation of Hypo solution:

S.No.	Burette Reading		Volume of hypo sol^{n}. used (ml)
	Initial Reading (ml)	Final Reading (ml)	
1.	0	14.7	14.7
2.			
3.			

ii) Titration of Hypo solution with water sample:

S.No.	Burette Reading		Volume of hypo sol^{n}. used (ml)
	Initial Reading (ml)	Final Reading (ml)	
1.	0	5.3	5.3
2.			
3.			

Calculations:

1. $N_1 V_1$ = $N_2 V_2$
   (K_2Cr_2O7) (Hypo)
   (1/100) X 10 = N_2 X ....
   N_2 (Normality of hypo. Solution) = ……..N

2. $N_2 V_2$ = $N_3 V_3$
   (Hypo) (Water solution)

…….X ……. = N_3 X 10

N_3 (Normality of water) = …….N

Strength of residual chlorine in water = N_3 X 35.5

= …… X 35.5

= …….. g/L

= …….. ppm

Result:

The strength of chlorine (residual) in water is 127.8 ppm.

Precautions:

1. Handle the apparatus with care.
2. Do not pipette out hot solutions.
3. There should be no air bubbles in burette.