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EXPERIMENT NO. 4

AIM:

To determine the strength of free chlorine in given water sample.

APPARATUS REQUIRED:

Burette, pipette, flask and glass rod.

CHEMICALS REQUIRED:

Given standard N/40 $CuSO_4$ solution to standardise sodium thiosulphate solution, potassium iodide solution (10%), N/40 Hypo solution ($Na_2S_2O_3$), starch solution (freshly prepared).

THEORY:

Chlorine is widely used for disinfection of potable and municipal water supplied to remove bacteria, fungus and other pathogenic micro organisms and for deodourisation, since it is a good oxidising agent and is cheaply available. Chlorination is done with help of bleaching powder or chlorine gas or chlorine dissolved in water in form of concentrated solution or with chloramines. The sterilizing action of chlorine is supposed to be due to its reaction with water producing hypochlorous acid and nascent oxygen both of which have powerful germicidal properties.

$$Ca(OCl)_2 + H_2O \rightarrow Ca(OH)_2 + Cl_2$$

$$Cl_2 + H_2O \rightarrow HOCl + HCl$$

$$H_2O + HOCl \rightarrow HCl + [O] + H_2O$$

$$HOCl + \text{germs} \rightarrow \text{germs are killed.}$$

However, excess of free chlorine in drinking water is undesirable and

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Chemical Reactions:

$$Ca(OCl)_2 + H_2O \rightarrow Ca(OH)_2 + Cl_2$$

$$Cl_2 + H_2O \rightarrow HOCl + HCl$$

$$H_2O + HOCl \rightarrow HCl + [O] + H_2O$$

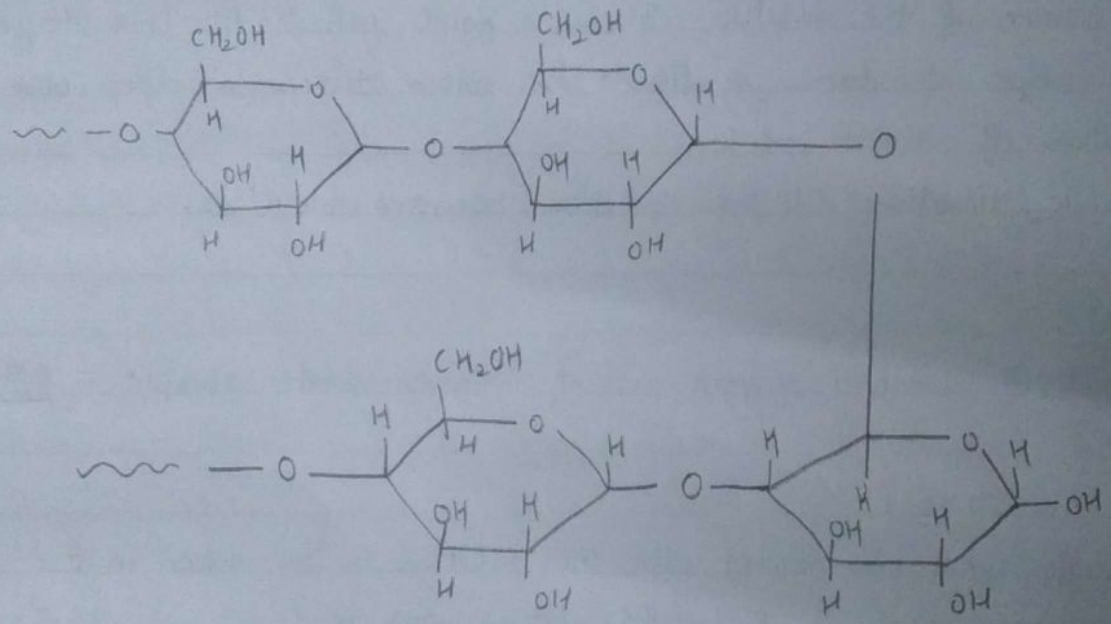
$HOCl$ + germs → germs are killed.

$$Cl_2 + 2KI \rightarrow 2KCl + I_2$$

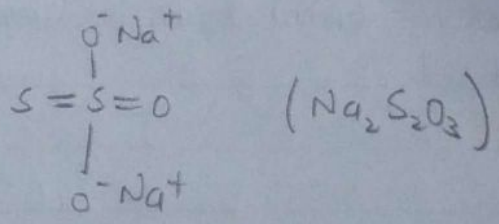
$$I_2 + 2Na_2S_2O_3 \rightarrow Na_2S_4O_6 + 2NaI$$

I_2 + starch indicator → Blue colour

Structure of Indicator: starch solution $(C_6H_{10}O_5)_n$



Hypo solution:



$(Na_2S_2O_3)$

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not only uppleasant for drinking but also injurious for human metabolism. Hence, the amount of free chlorine in municipal water is estimated prior to the domestic supply so as to make necessary adjustment in dose range accordingly. The principle involved is the estimation of free chlorine in water is that when a measured quantity is treated with excess of potassium iodide, the free chlorine present in the water oxidizes the corresponding amount of potassium iodide to iodine. The liberated iodine is estimated by titrating against standard hypo solution, using starch as indicator.

- $Cl_2 + 2KI \longrightarrow 2KCl + I_2$

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

  (Sodium thiosuphate) (Sodium tetrathionate)

  I_2 + Starch indicator $\longrightarrow$ Blue colour

- Starch is used an indicator.
- End point is obtained when soln. turns colourless from blue.
- Type of titrated involved is iodometric titration (type of potentiometric / redox titration).

PROCEDURE:

- Standardisation of hypo solution with given copper sulphate solution

1. Wash all the apparatus with the water.
2. Fill in the burette with the given hypo solution ($Na_2S_2O_3$).
3. Take 10 mL of given copper sulphate solution ($CuSO_4$) from pipette in conical flask.
4. Add 1mL of potassium iodide (KI) solution. The solution colour becomes dark yellow.

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OBSERVATIONS:

A) Standardisation of hypo solution with given $CuSO_4$ solution:

S.No.	Before adding starch			After adding 1-2 drop of starch			Total volume (mL) $=(V_1+V_2)$
	Initial	Final	V_1 (mL)	Initial	Final	V_2 (mL)	
1.	0	6.5	6.5	6.5	10.1	3.6	10.1
2.	10.1	16.6	6.5	16.6	20.2	3.6	10.1
3.	20.2	26.8	6.6	26.8	30.4	3.6	10.1

Concordant Reading = 10.1 mL

B) Titration of given water sample with hypo solution:

S.No.	Before adding starch			After adding starch			Total volume used $(=V_1+V_2)$ mL
	Initial	Final	V_1 (mL)	Initial	Final	V_2 (mL)	
1.	0	0.8	0.8	0.8	2.1	1.3	2.1
2.	2.1	2.9	0.8	2.9	4.2	1.3	2.1
3.	4.2	5.0	0.8	5.0	6.3	1.3	2.1

Concordant Reading = 2.1 mL

CALCULATIONS:

Vol. of Hypo soln. used for (A) = 10.1 mL
Vol. of Hypo soln. used for (B) = 2.1 mL
Vol. of $CuSO_4$ taken = 10 mL
Vol. of given water sample = 10 mL

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5. Titrate this solution with the hypo solution ($Na_2S_2O_3$) till faint yellow colour develops.
6. At this point, add 1-2 drops of starch as indicator. The soln. will turn blue in colour.
7. Titrate this faint yellow colour solution further with hypo to colourless.
8. Note the reading of burette when it becomes colourless. This is your end point.
9. Repeat this procedure to get the concordant reading for hypo soln.

• Titration of given water sample with hypo solution

1. Take 10mL of given water sample from pipette in a conical flask.
2. Add 1mL of potassium iodide (KI) solution. The solution colour becomes dark yellow.
3. Repeat the steps 3-9 of part-1.

RESULT:

The strength of free chlorine in given water sample is 181.04 ppm.

PRECAUTIONS:

1. Starch soln. was added at 99% completion of reaction which was identified by formation of faint yellow colour.
2. Lower meniscus of burette should be read.
3. The apparatus was rinsed with the solution to be taken in it.
4. The funnel must be removed before starting the titration.

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For A), $N_1 V_1 = N_2 V_2$

(Hyp soln.) ($CuSO_4$)

$$N_1 (10.1) = \frac{N}{40} (10)$$

$$N_1 = \frac{1}{4 \times 10.1} N = \underline{0.0247N}$$

Normality of hypo solution, $N_1 = \underline{0.0247N}$

For B) $N_3 V_3 = N_4 V_4$

(Hypo soln.) (given water sample)

$$(0.0247)(2.1) = N_4 (10)$$

$$N_4 = \underline{0.0051N}$$

Strength = Normality × Eq. weight of Cl_2

$$= 0.0051 \times \frac{71}{2} = 0.18104 g/L$$

$$= \underline{181.04 mg/L}$$

∴ Amount of free chlorine in given sample = $\underline{181.04 ppm}$