Date 4-04-2025

Expt. No. 6. Page No. 25

Object :- To determine the residual chlorine in a given water sample.

Apparatus and reagent :- Burette, pepette, Iodometric flask, Beakers, Droppers, Sodium thiosulphate ($Na_2S_2O_3 \cdot 5H_2O$), potassium dichromate ($K_2Cr_2O_7$), potassium iodide (KI), Sulphuric Acid (Conc. H_2SO_4), Acetic acid (CH_3COOH), ~~dichro~~ starch.

Theory :- Sterilized water is produced for drinking purposes by passing chlorine gas through the sample for complete removal of micro-organisms. The germicide action of chlorine gas produces hypochlorous acid (HOCl) and nascent oxygen [O]. If chlorine in water is beyond certain limit, the water will become unfit for drinking. It is therefore, necessary to determine the amount of free chlorine in water sample.

The analysis is based on the oxidation of potassium iodide (KI) by the free chlorine present in water. The liberated iodine is estimated iodometrically by titrating it against standard hypo solution using starch as indicator.

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

$$I_2 + \text{Starch} \longrightarrow \text{Starch + Iodo complex}$$

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

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Procedure :-

(A) Standardization of Hypo solution -

(i) Wash all the apparatus with distilled water.

(ii) Fill the burette with Hypo solution.

(iii) Pipette out 10 ml of $K_2Cr_2O_7$ solution becomes faint yellow.

(iv) Add to it 10 ml H_2SO_4 to maintain pH and 5 ml KI solution.

(v) Titrate it against hypo solution till the solution becomes faint yellow.

(vi) Now add few drops of freshly prepared starch indicator, a blue colour appears. Continue to add hypo solution drop by drop while constantly shaking till the blue color disappears, it indicates the end point.

(B) Titration of water sample with Hypo Solution -

(i) Fill the burette with hypo solution.

(ii) Take 10 ml of given water sample in a iodometric flask & add 3ml glacial acetic.

(iii) Add Acid to attain pH level 3-4. Shake the solution & add 5ml of KI solution.

(iv) Titrate the water sample with standard hypo Solution till the solution becomes faint yellow.

(v) Now add few drops of freshly prepared starch indicator, a blue colour appears, Continue to add hypo solution drop by drop while constantly shaking till the blue color disappear, it indicates the end point.

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Calculation:-

Titration between sample water with hypo solution

$N_1V_1 = N_2V_2$

$N_1 \times 50 = N/30 \times 4$

$N_1 = \frac{N}{50 \times 30} \times 4$

Here N_1 = Normality of sample water

N_2 = Normality of known Hypo

V_1 = Volume of sample water

V_2 = Volume of known Hypo.

Strength of free and residual chlorine.

Here N_3 = Normality of $K_2Cr_2O_7$

V_3 = Volume of $K_2Cr_2O_7$

N_4 = Normality of Hypo Solution

V_4 = Volume of ~~$K_2Cr_2O_7$~~ Hypo Solution.

$N_3V_3 = N_4V_4$

$N_3 = \frac{N_4V_4}{V_3}$

$N_3 = \frac{N}{50 \times 30} \times 4 \times \frac{9.0}{10}$

$N_3 = 0.0024$ ml

Strength = $N_3 \times$ Eq. weight $\times 1000$

$= 0.0024 \times 35.5 \times 1000$

$= 0.0852 \times 1000$

$= 85.2$ ppm Answer

Date

Expt. No. _____ Page No. 36

(vi) Note down the burette reading.

(vii) Repeat the titration till the concordant reading is obtained.

Observation

Titration between $K_2Cr_2O_7$ & hypo Solution

S. No.	Volume of $K_2Cr_2O_7$ (ml)	Volume of hypo Solution Initial (ml)	Final (ml)	Difference (ml)	Concordant reading (ml)
1.	10 ml	0.0	9.0	9.0	9.0
2.	10 ml	9.0	18.0	9.0	

Titration between ~~K_2~~ water and hypo solution.

S.No	Volume of water Sample (ml)	Volume of hypo Solution Initial (ml)	Final (ml)	Difference (ml)	Concordant reading (ml)
1.	50 ml	0	5	5	4.0
2.	50 ml	5	7.7	2.7	
3	50 ml	7.7	11.7	4.0	

Strength of free and residual chlorine is = ~~Nor~~

Normality $\times$ eq. weight of Chlorine $\times 1000$

$= 85.2$ ppm.

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Result :- Amount of free & residual chlorine in the given water sample = 85.2 ppm.

Precautions :-

1. Freshly prepared starch solution should be used
2. Distilled water should be used throughout the titration.
3. Starch solution be added just near end point.

Industrial Application :-

1. Chlorine determination is used to control chlorination's of domestic and industrial waste water.
2. Chlorine determination is important to avoid bad odor & change in taste of water.
3. It is determined in the swimming pools to avoid ill effects due to excess chlorination.

Viva Question

Q1 What are iodometric titrations?

Ans Iodometric titration is a type of redox titration that involves iodine (I_2) & is used to determine the concentration of oxidizing agents. It is an indirect titration method where an oxidizing agent reacts with excess iodine ions to produce iodine (I_2) which is then titrated with a reducing agent like sodium thiosulphate ($Na_2S_2O_3$)

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Q2 Differentiate between iodometric & iodimetric titration?

Ans Iodometic titration is an indirect method, where iodine is liberiated by an oxidizing agent & then titrated.

Iodimetric titration is a direct method, where iodine itself is used as a titrant to react with a reducing agent.

Q3 Diffentiate between primary & secondary solution.

Ans Primary solution are prepared from highly pure, stable compounds & have known concentration

Secondary solution have unknown or opportunities concentrations and must be standardized before we in titration.

Q4 Which type of indicator is used in this titration? Name it?

Ans Starch.

Q5 What is the equivalent weight of copper sulphate?

Ans equivalent weight of $CuSO_4$ = 79.75 gm/eq

$CuSO_4 \cdot 5H_2O$ (Hydrated. 249.80 gm/eq

Q6 Why freshly prepared starch solution is used in this titration?

Ans Because starch decompores over time, leading to inaccurate result.

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Q7 Why starch is added near the end point of the titration?

Ans Starch is added near the end point of titration, because it acts as an indicators for the presence of iodine. If we add starch in beginning then starch Iodine complex becomes too stable & may not dissociate.

Q8 Explain the reaction involved in this titration?

Ans When potassium iodide is added at pH (4 to 4.7) to copper sulphate solution, an equivalent amount of iodine is liberated & solution becomes brown.

$$2CuSO_4 + 4KI \longrightarrow 2K_2SO_4 + I_2 + Cu_2I_2$$

This liberated iodine is titrated with standard solution of sodium thiosulphate by using starch as an indicator

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

The liberated iodine absorbs on starch & given deep blue or violet colour.

$$\text{Starch} + I_2 \longrightarrow \text{Starch iodo complex (Violet / blue colour)}$$

from the above reaction it follows that

$$2CuSO_4 = I_2 = 2Na_2S_2O_3 \text{ or } Cu = I_2 = Na_2S_2O_3$$

Date

Expt. No. Page No. 40.

Q) Write chemical name & formula of Hypo Solution.

Ans Hypo solution is nothing but Sodium thiosulphate its chemical formula is $Na_2S_2O_3$

In aqueous form = $Na_2S_2O_3 \cdot 5H_2O$

Q) ~~How will you prepare N/30 CuSO4. Explain the principle of~~.

Q) Why pH should be maintained between 3 to 4 in this titration?

Ans Iodine remains stable & unreacted with hydroxide or oxygen.

Accurate stoichiometry in the redox reaction.

Reliable endpoint detection using starch.

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