

Lab Practical 3 (S2): Redox titration by Iodometric method (Determination of Chlorometric Degree in Bleach)

❖ Objectives

At the end of this lab practical, the student will be able to:

- **Recognize** the redox titration.
- **Carry out** an indirect titration using a complex experimental protocol.
- **Identify** the equivalence point (according to the different appear colors).
- **Calculate** the concentration of hypochlorite (ClO^-) present in commercial bleach.

Principle (primary objective)

To determine the chlorometric degree in commercial bleach.

I- Theoretical part

I-1 Definitions

➤ **Bleach:** is an aqueous solution of sodium hypochlorite (NaClO), used as a disinfectant or bleaching agent.

➤ **The chlorometric degree of a bleach solution (DC):**

The chlorometric degree of bleach is equal to the volume of dichlorine gas, measured under normal conditions (0C° and 1atm), necessary to make one liter of solution.



$$\text{DC}^\circ = C_{\text{ClO}^-} \times 22.4$$

(C_{ClO^-} is the molar concentration of hypochlorite expressed in mol/L)

- **Redox reaction:** Is a type of titration that examines the oxidation and reduction of certain chemical species. It is a transfer of electrons that happens from one atom to another (oxidation and reduction).
- When an atom donates electrons (reducing agent), there is always an atom that accepts electrons (oxidizing agent).
 - **Iodometric titration:** is an indirect redox titration used to determine the concentration of an oxidizing agent in a solution.

I-2 Titration reaction (iodometric titration)

The determination of hypochlorite (ClO^-) in bleach typically involves **two redox reactions:**

A-redox reaction 1: Reaction of Sodium hypochlorite (NaClO) with potassium iodide (KI).

hypochlorite ions ClO^- react with excess iodide ions I^- in the presence of sulfuric acid.

- Oxidation reaction 1:



- Reduction reaction 1:



Note: This step is crucial for the iodometric titration, where the I_2 generated is titrated with sodium thiosulfate.

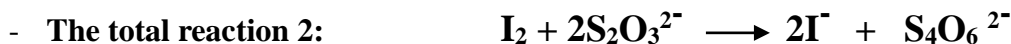
B- Redox reaction 2: Titration of Iodine (I_2) with Sodium Thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$).

The iodine formed in the **previous step** is titrated with a standard solution of **sodium thiosulfate**. The reaction that occurs is as follows.

- Oxidation reaction 2:

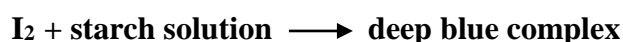


- Reduction reaction 2:



I-3 Equivalence point

A **starch (Amidon)** is often used as an **indicator** towards the end of the titration. Starch forms a **deep blue complex with iodine**, which **disappears** when all the iodine has reacted:



The disappearance of the blue color indicates the endpoint of the titration, signifying that all the I_2 has been reacted.

A- Calculation of ClO^- Normality:

➤ At the equivalence point for the 1st reaction:

$$N_{\text{Ox1}} \times V_{\text{Ox1}} = N_{\text{Red1}} \times V_{\text{Red1}} \longrightarrow \boxed{N(\text{I}_2) \times V(\text{I}_2) = N(\text{ClO}^-) \times V(\text{ClO}^-) \dots\dots(1)}$$

➤ At the equivalence point for the 2nd reaction:

$$N_{\text{Ox2}} \times V_{\text{Ox2}} = N_{\text{Red2}} \times V_{\text{Red2}} \longrightarrow \boxed{N(\text{S}_2\text{O}_3^{2-}) \times V(\text{S}_2\text{O}_3^{2-}) = N(\text{I}_2) \times V(\text{I}_2) \dots\dots(2)}$$

From (1) and (2) we have:
$$\boxed{N(\text{ClO}^-) \times V(\text{ClO}^-) = N(\text{S}_2\text{O}_3^{2-}) \times V(\text{S}_2\text{O}_3^{2-})}$$

$$\boxed{N(\text{ClO}^-) = N(\text{S}_2\text{O}_3^{2-}) \times V(\text{S}_2\text{O}_3^{2-}) / V(\text{ClO}^-)}$$

B- Calculation of ClO⁻ molar concentration:



Note: The bleach used is diluted (5%): $C_{\text{concentrated}}(\text{ClO}^-) = C(\text{ClO}^-) \times f$ (**f: dilution factor, f=20**)

C- Calculation of the chlorometric degree of a bleach solution (DC):

$$\text{DC}^\circ = C(\text{ClO}^-) \times 22.4$$

II- Practical part (Experimental protocol)

| Material | Products |
|---|---|
| <ul style="list-style-type: none"> - Erlenmeyer flask, Funnel. - Two graduated cylinders. - Two Graduated burettes. - Magnetic stirrer and magnetic stir bar. - Graduated pipette. | <ul style="list-style-type: none"> - Bleach (sodium hypochlorite) NaClO - Potassium iodide (KI) - Sulfuric acid (H₂SO₄) (0.1N) - Sodium thiosulfate (Na₂S₂O₃) (0.1N) - Starch |

➤ **Step1:**

- Measure 5 ml of a 5% dilute bleach (NaClO) solution and transfer it to an Erlenmeyer flask
- Add 7 ml of potassium iodide (KI) solution.
- Add 2 ml concentrated sulfuric acid (H₂SO₄).
- Allow mixture to stand under magnetic stirring ➡ The observed color turns brown, indicating the formation of iodine (I₂).

➤ **Step2:**

- Fill the burette with 0.1 N sodium thiosulfate solution (Na₂S₂O₃).
- Titrate the solution in (step 1) with sodium thiosulfate solution.
- When titration is almost complete (the solution takes on a straw-yellow color), add a few drops of starch (the color obtained is dark blue).
- Continue the titration until the dark blue color disappears.
- Note the volume of sodium thiosulfate solution at the equivalence point.

Report

The report must contain:

- A cover page according to the model.
- A detailed response to the questions at the end of the Lab Practical session.

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