BATNA2 University
Faculty of Natural and Life Sciences (L1)
Academic year 2023-2024

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 **<u>chlorometric degree</u>** 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.

$$ClO^- + 2H^+ + Cl^- \longrightarrow Cl_2\,(g) + H_2O$$

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

(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 **<u>transfer of electrons</u>** that happens from one atom to another **<u>(oxidation and reduction)</u>**.

- When an atom donates electrons **<u>(reducing agent)</u>**, there is always an atom that accepts electrons **<u>(oxidizing agent).</u>**

- **Iodometric titration:** is an **<u>indirect redox titration</u>** used to determine the concentration of an **<u>oxidizing agent</u>** 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 <u>(NaClO)</u> with potassium iodide <u>(KI).</u>

hypochlorite ions **ClO^-** react with excess iodide ions **I^-** in the presence of <u>sulfuric acid.</u>

- **Oxidation reaction 1**:

(Reducing agent) $2I^- \longrightarrow I_2 + 2é$

- **Reduction reaction 1:**

(Oxidizing agent) $ClO^- + 2H^+ + 2é \longrightarrow Cl^- + H_2O$

- **The total reaction 1:** $OCl^- + 2I^- + 2H^+ \longrightarrow I_2 + Cl^- + H_2O$

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 ($Na_2S_2O_3$).

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**:

(Reducing agent 2) $2S_2O_3^{2-} \longrightarrow S_4O_6^{2-} + 2é$

- **Reduction reaction 2:**

(Oxidizing agent 2) $I_2 + 2é \longrightarrow 2I^-$

- **The total reaction 2:** $I_2 + 2S_2O_3^{2-} \longrightarrow 2I^- + S_4O_6^{2-}$

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:

I_2 + starch solution $\longrightarrow$ **deep blue complex**

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:**

$N_{Ox1} \times V_{Ox1} = N_{Red1} \times V_{Red1} \longrightarrow$ $N(I_2) \times V(I_2) = N(ClO^-) \times V(ClO^-)$ ……..(1)

- **At the equivalence point for the 2nd reaction:**

$N_{Ox2} \times V_{Ox2} = N_{Red2} \times V_{Red2} \longrightarrow$ $N(S_2O_3^{2-}) \times V(S_2O_3^{2-}) = N(I_2) \times V(I_2)$ ……..(2)

From (1) and (2) we have: $N(ClO^-) \times V(ClO^-) = N(S_2O_3^{2-}) \times V(S_2O_3^{2-})$

$$N(ClO^-) = N(S_2O_3^{2-}) \times V(S_2O_3^{2-}) / V(ClO^-)$$

B- Calculation of ClO^- molar concentration:

$N(ClO^-) = x.\ C(ClO^-)$, $N(ClO^-) = 2.\ C(ClO^-)$, $ClO^- + 2H^+ + 2é \longrightarrow Cl^- + H_2O$

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

C- Calculation of the chlorometric degree of a bleach solution (DC): $DC° = C(ClO^-) \times 22.4$

II- Practical part (Experimental protocol)

Material	**Products**
- Erlenmeyer flask, Funnel. - Two graduated cylinders. - Two Graduated burettes. - Magnetic stirrer and magnetic stir bar. - Graduated pipette.	- Bleach (sodium hypochlorite) NaClO - Potassium iodide (KI) - Sulfuric acid (H_2SO_4) (0.1N) - Sodium thiosulfate ($Na_2S_2O_3$) (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 (H_2SO_4).
- Allow mixture to stand under magnetic stirring ⟶ The observed color turns brown, indicating the formation of iodine (I_2).

➢ **Step2:**

- Fill the burette with 0.1 N sodium thiosulfate solution ($Na_2S_2O_3$).
- 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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