

Experiment 5 redox titration using sodium thiosulphate

Experiment 5: Redox Titration Using Sodium Thiosulphate

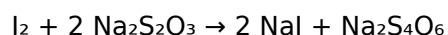
Introduction

Redox titrations are fundamental analytical techniques used to determine the concentration of oxidizing or reducing agents in a solution. Among the various titrants employed, sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) is widely used as a reducing agent due to its stability, ease of use, and specificity in certain redox reactions. This experiment involves titrating a known or unknown concentration of an oxidizing agent, such as iodine (I_2), with sodium thiosulphate. The process allows for precise quantification and understanding of redox chemistry principles, including oxidation states, electron transfer, and titration techniques.

Principles of the Redox Titration with Sodium Thiosulphate

Redox Reactions Involved

In this experiment, the primary reaction involves iodine being reduced by sodium thiosulphate:



Here, iodine (I_2) is reduced to iodide ions (I^-), while thiosulphate ($\text{S}_2\text{O}_3^{2-}$) is oxidized to tetrathionate ($\text{S}_4\text{O}_6^{2-}$). The reaction is a redox process where iodine acts as the oxidizing agent, and sodium thiosulphate as the reducing agent. The titration involves adding sodium thiosulphate to a solution containing iodine until the iodine is completely reduced, indicated by a color change.

Indicators Used

The endpoint of the titration is typically detected using a starch solution as an indicator: - When iodine is present, starch forms a blue-black complex. - As sodium thiosulphate reduces iodine to iodide, the blue-black color fades. - The titration is complete when the blue-black color disappears, indicating all iodine has been reduced.

Preparation for the Experiment

Materials Needed

1. Sodium thiosulphate solution (standardized)

2. Iodine solution (often prepared or purchased with known concentration)
3. Starch solution (as an indicator)
4. Distilled water
5. Conical flask
6. Burette
7. Funnels
8. Pipettes
9. Wash bottles
10. Clamp stand and holder

Preparation of Solutions

- Standardizing Sodium Thiosulphate: If not already standardized, sodium thiosulphate solution must be titrated against a primary standard such as potassium dichromate or potassium iodate to determine its exact concentration. - Iodine Solution: Usually prepared by dissolving a known mass of iodine in potassium iodide solution, which increases solubility.

Procedure of the Experiment

Step-by-Step Methodology

1. Prepare the iodine solution and record its concentration if not already known.
2. Using a pipette, transfer a known volume (e.g., 25 mL) of the iodine solution into a clean conical flask.
3. Add a few drops of starch solution to the flask. The solution will turn blue-black if iodine is present.
4. Fill the burette with sodium thiosulphate solution, ensuring no air bubbles are present in the nozzle.
5. Open the tap to allow the sodium thiosulphate to flow into the iodine solution slowly while swirling the flask constantly.
6. Continue titrating until the blue-black color just disappears, indicating all iodine has been reduced to iodide ions.
7. Record the volume of sodium thiosulphate used to reach the endpoint.
8. Repeat the titration at least three times to obtain consistent readings and calculate the average volume used.

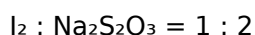
Calculations and Data Analysis

Determining the Concentration of the Unknown

The primary calculation involves using the titration data to find the concentration of the iodine solution or the unknown oxidizing agent. The key steps include: - Calculating the moles of sodium thiosulphate used:

$$\text{moles Na}_2\text{S}_2\text{O}_3 = \text{concentration (mol/L)} \times \text{volume (L)}$$

- Using the balanced reaction equation, relate moles of sodium thiosulphate to moles of iodine:



Therefore, moles of iodine = (moles $\text{Na}_2\text{S}_2\text{O}_3$) / 2 - Calculating the concentration of iodine:

Concentration of I_2 = (moles of I_2) / volume of iodine solution used (L)

Sample Calculation: Suppose 25 mL of iodine solution is titrated with 20 mL of 0.01 mol/L $Na_2S_2O_3$: - Moles of $Na_2S_2O_3$ = 0.01 mol/L $\times$ 0.020 L = 2.0×10^{-4} mol - Moles of I_2 = (2.0×10^{-4} mol) / 2 = 1.0×10^{-4} mol - Concentration of iodine = (1.0×10^{-4} mol) / 0.025 L = 4.0×10^{-3} mol/L

Applications of Sodium Thiosulphate Redox Titration

Common Uses

- Determining iodine content in pharmaceuticals and food products. - Analyzing oxidation states of various compounds. - Dechlorination of water supplies as sodium thiosulphate reacts with residual chlorine. - Titrating other oxidizing agents such as hydrogen peroxide, potassium permanganate, and halogens.

Precautions and Tips for Accurate Results

- Ensure all glassware is clean and free of impurities. - Standardize sodium thiosulphate solution regularly to maintain accuracy. - Add starch indicator carefully; excess can cause inaccuracies. - Titrate slowly near the endpoint to avoid overshooting. - Swirl the flask continuously during titration for uniform reaction.

Conclusion

Experiment 5 involving redox titration with sodium thiosulphate is a fundamental laboratory technique that reinforces understanding of oxidation-reduction reactions, titration methods, and quantitative analysis. Through careful preparation, precise titration, and accurate calculations, students can determine the concentration of oxidizing agents like iodine with high accuracy. This experiment not only elucidates core principles of redox chemistry but also highlights the importance of meticulous technique and data analysis in analytical chemistry. Its applications extend beyond the laboratory, impacting quality control, environmental analysis, and industrial processes where redox reactions are pivotal.

Experiment 5: Redox Titration Using Sodium Thiosulphate — A Comprehensive Review Redox titrations are fundamental procedures in analytical chemistry that allow the determination of the concentration of oxidizing or reducing agents in a solution. Among these, titrations involving sodium thiosulphate are particularly common due to its versatility, stability, and the ease with which its reactions can be monitored. This review provides an in-depth analysis of Experiment 5, which utilizes sodium thiosulphate in a redox titration, covering the principles, methodology, applications, and critical considerations.

Introduction to Redox Titration with Sodium Thiosulphate

Redox titration involves the transfer of electrons between species, characterized by oxidation states changing during the reaction. Sodium thiosulphate ($Na_2S_2O_3$) acts as a reducing agent and is frequently used as a titrant to quantify oxidizing agents like iodine (I_2), chlorates, and other oxidants. Key features of sodium thiosulphate in titrations: - It is a stable, water-soluble compound. - It reacts with iodine to

form tetrathionate ($\text{S}_4\text{O}_6^{2-}$) and iodide ions, facilitating precise endpoint detection. - Its solutions are relatively easy to prepare and standardize.

Principle of the Experiment

The core principle of the experiment revolves around the titration of a known or unknown concentration of an oxidizing agent (commonly iodine) with sodium thiosulphate. The fundamental reaction is:
$$\text{I}_2 + 2\text{Na}_2\text{S}_2\text{O}_3 \rightarrow 2\text{NaI} + \text{Na}_2\text{S}_4\text{O}_6$$
 This reaction proceeds with a clear and measurable change, enabling accurate determination of the analyte concentration. Overall steps: 1. Oxidize the analyte (e.g., potassium iodate or potassium dichromate) to liberate iodine. 2. Titrate the liberated iodine with sodium thiosulphate. 3. Use starch as an indicator to detect the endpoint accurately.

Preparation of Reagents

1. **Standard Sodium Thiosulphate Solution:** - Typically prepared by dissolving a known weight of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in distilled water. - Standardization against a primary standard such as potassium dichromate or iodine solution is essential for accurate results. 2. **Iodine Solution (if used):** - Can be prepared by dissolving a known mass of iodine in potassium iodide solution, or commercially available iodine solutions can be used. - The iodine solution is often standardized before use. 3. **Indicator Solution:** - Starch solution: Acts as an endpoint indicator, forming a blue-black complex with iodine, which disappears when iodine is reduced to iodide.

Experimental Procedure

1. **Sample Preparation:** Prepare the analyte solution, which could be a sample containing an oxidizing agent like potassium dichromate or an unknown substance.
2. **Oxidation Step:** If necessary, add an excess of potassium iodide (KI) to the sample. The oxidizing agent will oxidize iodide ions to iodine:
$$\text{Oxidant} + 2\text{I}^- \rightarrow \text{Reduced form} + \text{I}_2$$
3. **Extraction of Iodine:** The liberated iodine imparts a brownish color to the solution, indicating the presence of iodine.
4. **Titration:** Titrate the iodine solution with standardized sodium thiosulphate solution. Carefully add the titrant until the brown color begins to fade.
5. **Endpoint Detection:** Add a few drops of starch solution. The solution turns deep blue-black in the presence of iodine. Continue titration until the blue-black color just disappears, indicating the reduction of iodine to iodide.

Notes: - Perform titrations carefully to avoid overshooting the endpoint. - Repeat titrations to obtain concordant results (within ± 0.1 mL).

Calculations and Data Analysis

The primary goal is to determine the concentration of the unknown oxidizing agent based on the volume of sodium thiosulphate used. Step-by-step calculation: 1. Determine moles of sodium thiosulphate used:
$$\text{Moles Na}_2\text{S}_2\text{O}_3 = \text{Concentration} \times \text{Volume (L)}$$
 2. Calculate moles of iodine liberated: From the titration reaction, 1 mole of I_2 reacts with 2 moles of $\text{Na}_2\text{S}_2\text{O}_3$:
$$\text{Moles of I}_2 = \frac{\text{Moles of Na}_2\text{S}_2\text{O}_3}{2}$$
 3. Determine the amount of oxidizing agent in the sample:

Depending on the sample, relate the iodine produced to the original analyte via stoichiometry. 4. Concentration of the analyte: $\left[\frac{\text{Concentration of analyte}}{\text{volume of sample (L)}} \right]$ Example: Suppose 25.00 mL of sample requires 20.00 mL of 0.01 M $\text{Na}_2\text{S}_2\text{O}_3$ for titration: - Moles $\text{Na}_2\text{S}_2\text{O}_3$: $[0.01, \text{mol/L}] \times 0.020, \text{L} = 2.0 \times 10^{-4}, \text{mol}$ - Moles I_2 : $\left[\frac{2.0 \times 10^{-4}}{2} \right] = 1.0 \times 10^{-4}, \text{mol}$ - From the reaction, this corresponds to the amount of oxidant in the sample.

Applications of Sodium Thiosulphate Redox Titrations

The versatility of sodium thiosulphate makes it valuable across various fields: - Water treatment: Quantifying residual chlorine. - Food industry: Determining iodine content in iodized salt. - Pharmaceuticals: Assaying antioxidants or reducing agents. - Environmental analysis: Measuring levels of oxidants or pollutants.

Critical Aspects and Precautions

1. Standardization: - Always standardize sodium thiosulphate solutions before use to ensure accuracy. - Use primary standards like potassium dichromate or iodine solution for this purpose. 2. Endpoint Detection: - The starch indicator's color change must be observed carefully. - The endpoint should be approached slowly to avoid overshooting, which leads to inaccurate titration results. 3. Stability of Reagents: - Sodium thiosulphate solutions are prone to decomposition upon exposure to light and air; store in dark bottles and prepare fresh solutions periodically. 4. Interferences: - Presence of reducing agents or oxidants other than the analyte can interfere with titration. - Ensure purity of reagents and handle samples to minimize contamination. 5. Titration Technique: - Use a clean burette and pipette. - Perform titrations slowly near the endpoint for accuracy. - Repeat titrations for concordance.

Advantages and Limitations

Advantages: - Simple, cost-effective, and highly accurate with proper technique. - Suitable for large-scale or routine analyses. - The endpoint is clear with starch indicator. Limitations: - Sensitive to environmental factors like light and air. - Not suitable for samples containing substances that react with thiosulphate or iodine. - Requires careful standardization and technique.

Conclusion

Experiment 5 involving redox titration with sodium thiosulphate exemplifies a fundamental analytical technique with broad applicability across scientific disciplines. Its success hinges on meticulous preparation of reagents, precise titration techniques, and accurate calculations. Understanding the underlying chemistry, recognizing potential interferences, and adhering to best practices ensure reliable and reproducible results. This experiment not only reinforces core concepts of redox chemistry but also provides practical skills vital for analytical chemists, environmental scientists, and industry professionals. By mastering this titration process, students and practitioners can confidently analyze oxidizing agents in various matrices, contributing to quality control, environmental monitoring, and research advancements.

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Questions & Answers About experiment 5 redox titration using sodium thiosulphate

No	Question	Answer
1	What is the main objective of the redox titration using sodium thiosulphate in Experiment 5?	The main objective is to determine the concentration of an oxidizing agent, such as iodine, by titrating it with a standard sodium thiosulphate solution through a redox reaction.
2	Why is sodium thiosulphate commonly used as a titrant in redox titrations?	Sodium thiosulphate is used because it acts as a reliable reducing agent, has a stable concentration, and reacts quickly and completely with oxidizing agents like iodine, making it ideal for accurate titrations.
3	What are the typical indicators used in a redox titration involving sodium thiosulphate?	Starch solution is commonly used as an indicator because it forms a deep blue complex with iodine, allowing for precise detection of the endpoint when iodine is fully reduced.
4	How do you determine the endpoint in a sodium thiosulphate titration?	The endpoint is reached when the blue color from the iodine-starch complex disappears, indicating that all iodine has been reduced to iodide ions and the titration is complete.
5	What safety precautions should be taken during a redox titration with sodium thiosulphate?	Safety precautions include wearing gloves and goggles, handling chemicals carefully to avoid spills, working in a well-ventilated area, and properly disposing of chemical waste to prevent environmental contamination.

redox titration, sodium thiosulphate, oxidation-reduction, titration method, iodine titration, reducing agent, oxidizing agent, endpoint detection, titrant, analyte

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