


Experiment 9 Prelaboratory Assignment A

Volumetric Analysis



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Experiment 9

A Volumetric Analysis


A titrimetric analysis requires the careful addition of titrant.

- To prepare and standardize a sodium hydroxide solution
- To determine the molar concentration of a strong acid

OBJECTIVES

The following techniques are used in the Experimental Procedure:

TECHNIQUES



A chemical analysis that is performed primarily with the aid of volumetric glassware (e.g., pipets, burets, volumetric flasks) is called a **volumetric analysis**. For a volumetric analysis procedure, a known quantity of a carefully measured amount of one substance reacts with a to-be-determined amount of another substance with the reaction occurring in aqueous solution. The volumes of all solutions are carefully measured with volumetric glassware.

The known amount of the substance for an analysis is generally measured and available in two ways:

- As a **primary standard**—An accurate mass (and thus, moles) of a solid substance is measured on a balance, dissolved in water, and then reacted with the substance being analyzed.
- As a **standard solution**—A measured number of moles of substance is present in a measured volume of solution, generally expressed as the molar concentration (or molarity) of the substance. A measured volume of the standard solution then reacts with the substance being analyzed.

The reaction of the known substance with the substance to be analyzed, occurring in aqueous solution, is generally conducted by a titration procedure.

The titration procedure requires a buret to dispense a liquid, called the **titrant**, into a flask containing the **analyte** (Figure 9.1a, page 134). For the acid-base titration studied in Part B of this experiment, the titrant is a standard solution of sodium hydroxide and the analyte is an acid.

INTRODUCTION

Primary standard: a substance that has a known high degree of purity, a relatively large molar mass, is nonhygroscopic, and reacts in a predictable way

Standard solution: a solution having a very well known concentration of a solute

Experiment 9 Prelaboratory Assignment: A Volumetric Analysis

Volumetric analysis is a critical technique in quantitative chemistry, allowing chemists to determine the concentration of an unknown solution through titration. In Experiment 9, the prelaboratory assignment focuses on the principles, procedures, and calculations involved in volumetric analysis, emphasizing the importance of accurate measurement and careful technique. This article aims to provide a comprehensive overview of the concepts surrounding volumetric analysis, including the necessary preparations, the equipment required, and the calculations performed during the experiment.

Understanding Volumetric Analysis

Volumetric analysis, often referred to as titration, is a method used to determine the concentration of a solute in a solution by reacting it with a solution of known concentration. This technique is predicated on the precise measurement of volumes, and it typically involves the use of a burette, a pipette, and a flask.

Principles of Volumetric Analysis

The fundamental principle behind volumetric analysis is based on stoichiometry. When two solutions react, the relationship between their concentrations and the volumes used can be described by the balanced chemical equation. The point at which the reaction is complete is known as the equivalence point, which is often indicated by a color change due to an indicator.

Key concepts include:

1. Molarity (M): The number of moles of solute per liter of solution.
2. Stoichiometric Ratios: The ratio derived from the balanced chemical equation, which allows for the calculation of the amount of reactant or product involved in the reaction.
3. Titration Curve: A graph plotting the pH of the solution against the volume of titrant added, which helps visualize the equivalence point.

Preparing for the Experiment

Proper preparation is vital for the success of a volumetric analysis experiment. This includes understanding the chemicals involved, preparing solutions, and familiarizing oneself with the equipment.

Safety Precautions

Before beginning any laboratory work, safety precautions must be taken to ensure a safe working environment. Some important safety measures include:

- Wearing safety goggles and a lab coat to protect against splashes.
- Handling all chemicals with care, especially strong acids and bases.
- Ensuring proper ventilation when working with volatile substances.

Materials Required

The following materials are typically required for a volumetric analysis experiment:

- Burette: For dispensing the titrant accurately.
- Pipette: For measuring a specific volume of the analyte.
- Erlenmeyer Flask: To contain the solution being titrated.
- Indicator: A substance that changes color at or near the equivalence point.
- Standard Solution: A solution of known concentration used for titration.
- Distilled Water: For rinsing equipment and diluting solutions.

Preparing Solutions

1. Standard Solution Preparation:

- Accurately weigh the required amount of solute (e.g., sodium chloride or potassium permanganate).
- Dissolve it in a known volume of distilled water in a volumetric flask, ensuring the solution is homogeneous.

2. Dilution of Solutions:

- Use the dilution formula $C_1V_1 = C_2V_2$ to prepare diluted solutions, where C is concentration and V is volume.
- Ensure thorough mixing after dilution.

Conducting the Titration

Titration involves a systematic approach to ensure accurate results. The following steps outline the titration process:

Setting Up the Apparatus

1. Clean and Rinse Equipment:

- Rinse the burette with distilled water and then with the titrant solution to avoid contamination.
- Rinse the pipette with the analyte solution to ensure accurate measurement.

2. Filling the Burette:

- Fill the burette with the titrant solution, ensuring there are no air bubbles in the nozzle.
- Record the initial volume.

3. Pipetting the Analyte:

- Use the pipette to transfer a measured volume of the analyte to the Erlenmeyer flask.
- Add a few drops of the chosen indicator to the solution.

Titration Procedure

1. Start Titration:

- Open the burette to allow the titrant to flow into the analyte solution.

- Swirl the flask gently to mix the solutions.

2. Approaching the Endpoint:

- As you near the endpoint (indicated by a color change), add the titrant dropwise.
- Carefully observe the solution for any permanent color change.

3. Determining the Endpoint:

- The endpoint is reached when a stable color change persists. Record the final burette reading.

4. Repeat:

- Perform multiple titrations to ensure reliability and accuracy, ideally three trials, and calculate the average volume of titrant used.

Calculations and Analysis

Once the titration is complete, calculations are necessary to determine the concentration of the unknown solution.

Calculating Molarity

The molarity of the analyte can be calculated using the formula derived from the stoichiometry of the reaction:

$$M_1V_1 = M_2V_2$$

Where:

- M_1 = molarity of the titrant

- V_1 = volume of the titrant used
- M_2 = molarity of the analyte (unknown)
- V_2 = volume of the analyte used

To find M_2 , rearrange the equation:

$$M_2 = \frac{M_1 V_1}{V_2}$$

Error Analysis

It is crucial to evaluate potential sources of error in volumetric analysis, which can include:

- Equipment Calibration: Ensure all glassware is calibrated correctly.
- Human Error: Inaccurate reading of the burette or pipette can lead to discrepancies.
- Environmental Factors: Variations in temperature can affect solution volume.

Consider documenting any observed anomalies during the experiment to address them in the discussion section of the lab report.

Conclusion

Volumetric analysis is an indispensable method in quantitative chemistry, providing precise measurements of solute concentrations through careful titration techniques. The successful completion of Experiment 9 requires a thorough understanding of the underlying principles, meticulous preparation, and accurate execution of the titration process. By adhering to safety protocols and proper laboratory techniques, students can gain valuable insights into the quantitative aspects of chemistry, laying a foundation for future scientific endeavors. Understanding these concepts will not only enhance

laboratory skills but also foster a deeper appreciation for the analytical methods employed in chemical research and industry.

Frequently Asked Questions

What is the primary objective of Experiment 9 in volumetric analysis?

The primary objective of Experiment 9 is to determine the concentration of an unknown solution through titration, using a standard solution of known concentration.

What materials are typically required for a volumetric analysis experiment?

Common materials include a burette, pipette, volumetric flask, standard solution, the analyte solution, a white tile, and a pH indicator or phenolphthalein.

Why is it important to use a standard solution in volumetric analysis?

A standard solution provides a reliable reference point for the concentration of the analyte, allowing for accurate calculations of the unknown concentration through titration.

What safety precautions should be taken during Experiment 9?

Safety precautions include wearing lab coats and safety goggles, handling chemicals with care, and ensuring proper disposal of waste materials to prevent contamination.

How is the end point of a titration determined in volumetric analysis?

The end point of a titration is determined by observing a color change in the indicator used, which signifies that the reaction between the titrant and analyte is complete.

What calculations are performed after completing the titration in Experiment 9?

Calculations typically involve determining the molarity of the unknown solution using the volume of titrant used, the concentration of the standard solution, and the stoichiometry of the reaction.

What is the significance of using a white tile during the titration process?

A white tile is used to provide a contrasting background, making it easier to observe the color change of the indicator during the titration process.

What common mistakes should be avoided during volumetric analysis?

Common mistakes include not rinsing glassware properly, misreading the meniscus, adding titrant too quickly, and failing to record measurements accurately.

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