

Gravimetry

Gravimetric Analysis: Principle and Steps

Principle:

- Gravimetric analysis is a quantitative method that determines the amount of an analyte based on its mass.
- The analyte is isolated, typically as a precipitate, which is then weighed to calculate the analyte's concentration.

Steps in Gravimetric Analysis:

1. Sample Preparation:

- Weigh a known amount of the sample and dissolve it in a suitable solvent (e.g., distilled water).
- Filter to remove impurities if necessary.

2. Precipitation:

- Add a precipitating agent that reacts selectively with the analyte to form an insoluble precipitate.

3. Digestion:

- Allow the precipitate to settle, often by heating or standing, to improve purity and particle size.

4. Filtration:

- Filter the precipitate to separate it from the solution using appropriate filter paper.

5. Washing and Drying:

- Wash the precipitate to remove impurities, then dry it in an oven or desiccator to remove moisture.

6. Weighing:

- Weigh the dried precipitate using an analytical balance.

7. Calculation:

- Use the mass of the precipitate and the stoichiometry of the reaction to determine the analyte's concentration.

This concise process ensures the accurate determination of an analyte based on its mass.

Purity of the precipitate: co-precipitation and post precipitation

- The purity of the precipitate is essential in gravimetric analysis, as impurities can significantly affect the accuracy of results.

Two common sources of impurities are co-precipitation and post-precipitation:

A. Co-precipitation

- Co-precipitation occurs when impurities or other ions in the solution precipitate along with the target analyte during the precipitation process.

This can happen due to:

a. Surface Adsorption:

- Impurities adsorb onto the surface of the precipitate particles, leading to contamination.

b. Occlusion and Entrainment:

- Impurities become trapped within or between the precipitate particles during their formation.

c. Mixed Crystal Formation:

- Impurities are incorporated into the crystal lattice of the precipitate, resulting in mixed crystals.

Minimizing Co-precipitation:

- Optimize precipitation conditions (temperature, pH, and concentration) to favor pure precipitate formation.
- Use a selective precipitating agent that reacts only with the target analyte.
- Perform digestion by heating or aging the precipitate to improve its purity and particle size.

B. Post-precipitation

- Post-precipitation happens when the initial precipitate partially dissolves, and the analyte re-precipitates with impurities.
- This often occurs due to changes in the solution's pH, temperature, or concentration during the analysis process.

Minimizing Post-precipitation:

- Maintain constant conditions (temperature, pH) throughout the analysis to prevent dissolution and re-precipitation.
- Wash the precipitate thoroughly with a suitable solvent to remove soluble impurities.
- Dry the precipitate gently, avoiding high temperatures that may cause decomposition or reactions with impurities.

Estimation of Barium Sulfate (BaSO_4) by Gravimetric Analysis

Steps:

1. Sample Preparation:

- Weigh the sample containing barium ions (Ba^{2+}) and dissolve in distilled water or dilute HCl.
- Filter the solution to remove insoluble impurities.

2. Precipitation:

- Add sodium sulfate (Na_2SO_4) to the solution.
- Barium ions react with sulfate ions to form barium sulfate precipitate:

$$\text{Ba}^{2+}(\text{aq}) + \text{SO}_4^{2-}(\text{aq}) \rightarrow \text{BaSO}_4(\text{s})$$

3. Digestion:

- Let the precipitate settle, and optionally heat the solution to improve the precipitate's purity and particle size.

4. Filtration:

- Filter the precipitate using suitable filter paper or apparatus.

5. Washing and Drying:

- Wash the precipitate with distilled water to remove contaminants.
- Transfer the precipitate to a pre-weighed crucible and dry in an oven or desiccator.

6. Weighing:

- Weigh the dried precipitate and subtract the weight of the empty crucible to obtain the mass of BaSO_4 .

7. Calculation:

- Calculate the percentage of barium using:

$$\% \text{Ba} = \left(\frac{\text{Mass of BaSO}_4 \times \text{Molar mass of Ba}}{\text{Weight of sample} \times \text{Molar mass of BaSO}_4} \right) \times 100$$

This streamlined process ensures accurate determination of barium in the sample via gravimetric analysis.

Diazotization titration

Introduction

- Diazotization titration, also known as diazo coupling titration, is a versatile and widely used method for the quantitative determination of **primary aromatic amines**.
- The technique is based on the reaction of aromatic amines with **nitrous acid (HNO₂)** to form **diazonium salts**, which can further couple with suitable coupling agents to form highly colored azo compounds.
- The colored compounds can be analyzed **spectrophotometrically** or **visually** using indicators.

Basic Principles:

- In diazotization titration, the **primary aromatic amine** reacts with **nitrous acid**, which is generated *in situ* by the reaction of **sodium nitrite (NaNO₂)** and a strong acid (e.g., HCl or H₂SO₄).
- The resulting **diazonium salt** is highly reactive and couples with a **phenolic or naphtholic compound** to form a colored **azo dye**.
- The formation of the azo dye is used as an **endpoint** in the titration.

Basic Reactions Involved:

1. Formation of nitrous acid:



2. Diazotization reaction:



(Ar represents an aromatic group)

3. Coupling reaction:



(Ar' represents another aromatic group, usually the coupling agent)

Methods

1. Direct Titration Method

- The primary aromatic amine is titrated directly with a **standard solution of sodium nitrite** in the presence of **excess acid**.
- The endpoint is determined using a **suitable indicator**.

2. Back Titration Method (Indirect Method)

- An **excess of sodium nitrite** is added to the primary aromatic amine in the presence of excess acid.
- The unreacted nitrite is then **back-titrated** with a standard solution of another compound, such as **potassium iodate** or **ammonium thiocyanate**.

Applications:

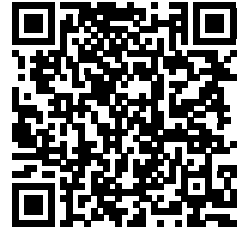


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