

Qualitative Analysis

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PURPOSE: Using an analytical scheme, four cations (Ag^+ , Pb^{2+} , Al^{3+} and Ni^{2+}) are separated and identified. Then this scheme is applied to identify cations in an unknown mixture.

Required Reading

- Ref. (1), section 4.2, subsections "The Key Event: Formation of a Solid from Dissolved Ions" and "Predicting Whether a Precipitate Will Form"; section 4.4, subsection "Using Oxidation Numbers to Monitor Electron Charge"

Introduction

Qualitative analysis is a set of methods in analytical chemistry that addresses the question "What is in a sample?" In inorganic chemistry, qualitative analysis is based on the fact that different ions have different chemical properties and show specific changes upon addition of a reagent. Using precipitation reactions, cations are sequentially removed from the mixture. For example, if a solution contains various metal ions, the addition of HCl would specifically precipitate out Ag^+ , Pb^{2+} , and Hg_2^{2+} . If a precipitate (ppt) can only contain one cation it may serve as a proof for the presence of that cation. If a precipitate contains more than one ion, it can be re-dissolved and in further steps, the cations can be separated and their presence or absence confirmed. These steps may include acid-base reactions, complexation reactions, redox reactions and precipitation reactions.

To separate the precipitate from the balance of the solution, we can employ filtration, centrifugation or decantation. Filtration utilizes a filter medium (usually filter paper) to separate the solid from the liquid. Centrifugation uses centrifugal force for separation of mixtures. The denser components of the mixture migrate away from the axis of the centrifuge while the less dense components migrate towards the axis. The solution, which lies above the solid and is called the supernatant, is simply poured (decanted) off leaving the solid behind.

Because much of qualitative analysis involves selective precipitation and then separation of the two phases, it is convenient to represent an analytical scheme in the form of a flowchart. Figure 1 shows a simple flowchart for separating a known mixture of Ag^+ , Pb^{2+} , Hg_2^{2+} , and Cu^{2+} ions. The addition of each reagent (presented in order from top to bottom) produces a solid phase and an aqueous phase that can be separated by centrifugation or filtration. The flowchart translates to the following steps: (1) add 6 M HCl to precipitate AgCl , PbCl_2 , and Hg_2Cl_2 from the mixture; (2) centrifuge sample to yield a solid pellet and a supernatant;

(3) remove the supernatant and add to it NH_3 to produce a blue complex ion, $[\text{Cu}(\text{NH}_3)_4]^{2+}$; (4) To the ppt, add hot deionized water and warm it to dissolve PbCl_2 ; (5) centrifuge; (6) remove supernatant and add to it acetic acid and potassium chromate to precipitate PbCrO_4 ; (7) Add NH_3 to the ppt of AgCl and Hg_2Cl_2 . A black ppt of $\text{Hg}(\text{NH}_2)\text{Cl}$ and $\text{Hg}(\text{l})$ and a complex ion ($[\text{Ag}(\text{NH}_3)_2]^+$) will form; (8) centrifuge; (9) remove the supernatant and add HNO_3 to re-precipitate AgCl . Compare the chemical equations below the flowchart with the steps in the procedure.

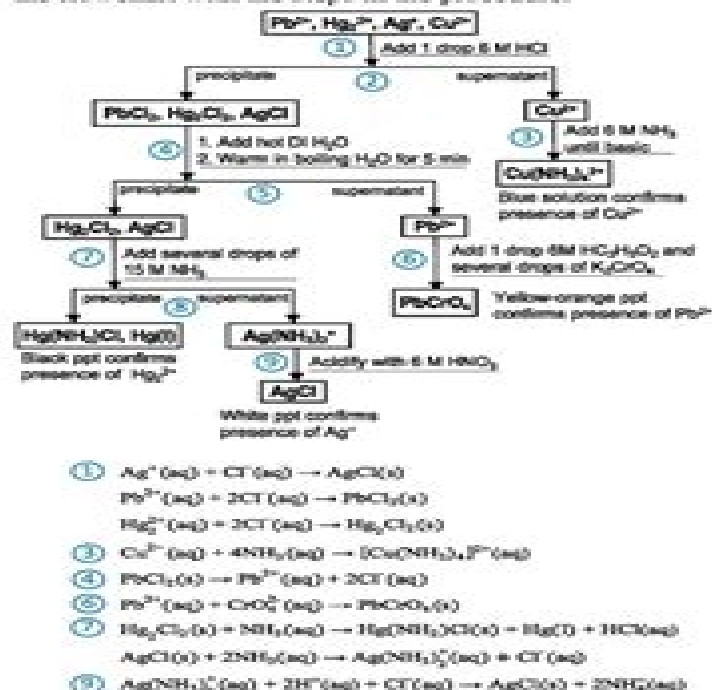


Figure 1. Flowchart and net ionic equations for the separation of Pb^{2+} , Hg_2^{2+} , Ag^+ and Cu^{2+}

In any qualitative analysis it is important to run your analytical scheme first on "known" samples that contain specific ions of interest, and then on "unknown" samples in order to test your ability to use the procedure to analyze a solution of unknown composition. In this way, you can observe first-hand how a particular ion behaves when a certain reagent is added and make comparisons with the behavior of your unknown sample.

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Table of Contents Qualitative Analysis An Introduction To Equilib

1. Understanding the eBook Qualitative Analysis An Introduction To Equilib
 - The Rise of Digital Reading Qualitative Analysis An Introduction To Equilib
 - Advantages of eBooks Over Traditional Books
2. Identifying Qualitative Analysis An Introduction To Equilib
 - Exploring Different Genres
 - Considering Fiction vs. Non-Fiction
 - Determining Your Reading Goals
3. Choosing the Right eBook Platform
 - Popular eBook Platforms
 - Features to Look for in an Qualitative Analysis An Introduction To Equilib
 - User-Friendly Interface
4. Exploring eBook Recommendations from Qualitative Analysis An Introduction To Equilib
 - Personalized Recommendations
 - Qualitative Analysis An Introduction To Equilib User Reviews and Ratings
 - Qualitative Analysis An Introduction To Equilib and Bestseller Lists

5. Accessing Qualitative Analysis An Introduction To Equilib Free and Paid eBooks
 - Qualitative Analysis An Introduction To Equilib Public Domain eBooks
 - Qualitative Analysis An Introduction To Equilib eBook Subscription Services
 - Qualitative Analysis An Introduction To Equilib Budget-Friendly Options
6. Navigating Qualitative Analysis An Introduction To Equilib eBook Formats
 - ePub, PDF, MOBI, and More
 - Qualitative Analysis An Introduction To Equilib Compatibility with Devices
 - Qualitative Analysis An Introduction To Equilib Enhanced eBook Features
7. Enhancing Your Reading Experience
 - Adjustable Fonts and Text Sizes of Qualitative Analysis An Introduction To Equilib
 - Highlighting and Note-Taking Qualitative Analysis An Introduction To Equilib
 - Interactive Elements Qualitative Analysis An Introduction To Equilib
8. Staying Engaged with Qualitative Analysis An Introduction To Equilib
 - Joining Online Reading Communities
 - Participating in Virtual Book Clubs
 - Following Authors and Publishers Qualitative Analysis An Introduction To Equilib
9. Balancing eBooks and Physical Books Qualitative Analysis An Introduction To Equilib
 - Benefits of a Digital Library
 - Creating a Diverse Reading Collection Qualitative Analysis An Introduction To Equilib
10. Overcoming Reading Challenges
 - Dealing with Digital Eye Strain
 - Minimizing Distractions
 - Managing Screen Time
11. Cultivating a Reading Routine Qualitative Analysis An Introduction To Equilib
 - Setting Reading Goals Qualitative Analysis An Introduction To Equilib
 - Carving Out Dedicated Reading Time
12. Sourcing Reliable Information of Qualitative Analysis An Introduction To Equilib
 - Fact-Checking eBook Content of Qualitative Analysis An Introduction To Equilib
 - Distinguishing Credible Sources
13. Promoting Lifelong Learning

- Utilizing eBooks for Skill Development
- Exploring Educational eBooks

14. Embracing eBook Trends

- Integration of Multimedia Elements
- Interactive and Gamified eBooks

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