Experiment 25

QUALITATIVE ANALYSIS

MATERIALS: 0.1 M solutions of Pb\(^{2+}\), Ag\(^+\), Fe\(^{3+}\), Ni\(^{2+}\), and Al\(^{3+}\); a solution that is 0.10 M in all five ions; 6 M HCl; 3 M NaOH; 1.5 M K\(_2\)CrO\(_4\); 1% Dimethylglyoxime (DMG) in aqueous ethanol; 15 M NH\(_3\); 1 M KSCN; unknowns; red and blue litmus paper; test tubes; centrifuge; Bunsen burner.

PURPOSE: The purpose of this experiment is to identify the metal ions present in an unknown solution.

LEARNING OBJECTIVES: By the end of this experiment, the student should be able to demonstrate the following proficiencies:
1. Conduct chemical tests to determine the presence or absence of specific ions in a solution.
2. Predict the product of a precipitation reaction.
3. Read and follow a flow chart.
4. Construct a flow chart from the results of a given series of chemical reactions.
5. Summarize the results and observations from an experiment into a short laboratory report.

DISCUSSION:

There are two types of problems that are of interest to analytical chemists. One is qualitative analysis, which is the determination of the presence or absence of a given species in a sample. The other is quantitative analysis, which is the determination of how much of a species is present in a sample.

This experiment involves the qualitative analysis of solutions containing one or more metal cations. Several specific reactions will be carried out on solutions that will either separate the ions from each other or confirm the presence of one particular ion. The reactions that confirm the presence of a particular ion involve the creation of visible products such as precipitates or colored species. A reaction that results in a precipitation can also be used to separate ions from the solution.

Qualitative analysis is relatively simple when a solution is known to contain only one ion. For example, suppose a solution contains either of the ions A\(^{2+}\) or B\(^+\). It is known that A\(^{2+}\) reacts with C\(^-\) to form a yellow precipitate while B\(^+\) combines with C\(^-\) to form a green precipitate:

\[
\begin{align*}
A^{2+} + C^- & \rightarrow \text{yellow precipitate} \\
B^+ + C^- & \rightarrow \text{green precipitate}
\end{align*}
\]

Identifying which ion is present in solution only requires addition of C\(^-\) to the solution and observation of the resultant precipitate color. Difficulty arises if the solution contains both ions. In this case, the combination might result in a slightly lighter green precipitate, making it difficult to determine if the solution contained only B\(^+\) or both ions. The presence of B\(^+\) interferes with the test for A\(^{2+}\) and vice versa.

The strategy for investigating solutions with multiple ions is to first find chemical reactions that will separate the ions. Once the separation has been accomplished, the ions can be tested and identified individually. For example, in the case of the A\(^{2+}\) and B\(^+\) mixture, it is found that addition of base will result in the formation of A(OH)\(_2\) which is insoluble and precipitates from solution. In contrast, B\(^+\) does not react with the base and remains as an ion in solution.

\[
\begin{align*}
A^{2+} + 2 \text{OH}^- & \rightarrow \text{A(OH)\(_2\) precipitate} \\
B^+ + \text{OH}^- & \rightarrow \text{no reaction}
\end{align*}
\]

Therefore, treating the unknown solution with sodium hydroxide can result in one of the following two situations:

1. No precipitate: The conclusion is that A\(^{2+}\) is not present. Confirm the presence or absence of B\(^+\) by adding C\(^-\) to the solution and looking for the green precipitate.
2. Presence of a precipitate: Separate the precipitate (possibly containing $A^{2+}$) from the supernatant (possibly containing $B^+$) by centrifuging the test tube and decanting (pouring off) the supernatant, which is the liquid above the precipitate. The supernatant is then tested for the presence $B^+$ as described above. The precipitate should also be tested to verify that it contains $A^{2+}$ and not some other impurity that precipitates in base. To do this, dissolve the precipitate in neutral or acidic solution, add $C^-$, and look for the yellow precipitate.

These possibilities are illustrated in the flow chart shown below.

![Flow Chart](image-url)

Figure 1. Flow Chart for Separation and Identification of $A^{2+}$ and $B^+$

The lab work for this experiment will be performed over three lab periods. During the first two periods, conduct the tests described in the procedure with the known solutions provided. Start with the single ion solutions. Use the observations from these tests to devise a qualitative analysis scheme that will allow for separation of the ions and identification of the ions present in an unknown. Test the flow chart by using the available solution that contains all five ions. Carefully record observations for each reaction. While carrying out these reactions, take specific note of which reactions will separate the ions and which reactions give distinctive products which allow for the identification of specific ions. For example, there may be only one bright crimson solid produced. Also, note which ions interfere with the detection of other ions. Making up known mixtures of ions to test may be helpful in confirming your observations. Once a successful scheme has been developed, determine which specific ions are present in an unknown solution.

**Experiment 25 Time Line**

<table>
<thead>
<tr>
<th>Week</th>
<th>Prior to Lab</th>
<th>During Lab</th>
<th>Post Lab</th>
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<tbody>
<tr>
<td>Week 1</td>
<td>Read the Discussion</td>
<td>Part A – Individual Ions</td>
<td>Develop initial flow chart</td>
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<td>Answer In-lab questions</td>
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<tr>
<td>Week 2</td>
<td>Prepare for lab</td>
<td>Finish Part A</td>
<td>Revise and finalize your</td>
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<td></td>
<td>Part B – Five Ion Solution</td>
<td>flow chart; Have your</td>
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<td>instructor check your</td>
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<td></td>
<td></td>
<td></td>
<td>flow chart</td>
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<td>Week 3</td>
<td>Prepare for lab</td>
<td>Part C – Unknown Solution</td>
<td>Prepare the lab summary</td>
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PROCEDURE:

The chemical reactions (tests) used in this experiment are described below. The reagents are in small dropper bottles. Be careful not to contaminate any of the reagents.

Carefully record observations for each reaction in the appropriate tables. These observations should include a detailed description of the appearance of any precipitates (i.e., gelatinous, wispy, deep red, voluminous, etc.) and any color changes.

Dispose of all solutions in the proper waste containers.

Part A. Qualitative Tests for the Individual Ions

Test I. Chloride Test
1. Clean 5 test tubes. Use a test tube brush to remove any solid material. Rinse the tubes well with distilled water.
2. Place 10 drops of each individual metal solution (Pb^{2+}, Ag^{+}, Fe^{3+}, Ni^{2+}, Al^{3+}) into its own test tube. Label the test tubes.
3. Add 5 drops of 6 M HCl to each test tube. Mix the solutions. Record your observations in the Data Section.
4. a. If a precipitate forms, mix the solution well to allow complete reaction and centrifuge for 5 minutes. Make sure the centrifuge is balanced before spinning your sample. Continue with Step 5.
   b. If no precipitate forms, dispose of these solutions in the proper waste container.
5. After centrifuging, check the supernatant (the liquid above the precipitate) for complete precipitation by adding a few more drops of 6 M HCl. If more precipitate forms, centrifuge again for 2 minutes.
6. Wash the precipitate twice with 10 drops of distilled water. To wash the precipitate, decant (pour off) the supernatant, add distilled water, and mix well. Centrifuge after each addition of water and decant. Save the precipitate for use in Test II.

Test II. Hot Water/K_{2}CrO_{4} Test
A. Hot Water Test
1. Add 15 drops of distilled water to each precipitate from Test I.
2. Heat the test tubes in a boiling water bath for 5 to 10 minutes. While heating, mix the contents of the test tube often. Did any of the precipitates dissolve? Record your observations. If the ion did not give a precipitate in Test I, put an X in the corresponding box.
B. K_{2}CrO_{4} Test
3. a. If a precipitate is still present after heating, centrifuge and save the decanted supernatant. Add 2 drops of K_{2}CrO_{4} solution to the hot supernatant.
   b. If the precipitate dissolved upon heating, add 2 drops of K_{2}CrO_{4} to the hot liquid.
4. Record your observations.

Test III. NaOH Test
1. Clean your test tubes. Add 10 drops of each individual metal solution (Pb^{2+}, Ag^{+}, Fe^{3+}, Ni^{2+}, Al^{3+}) into its own test tube. Label the test tubes.
2. To each test tube, add 1 drop of 3 M NaOH solution, mix well, and note any changes. Repeat this process until you have added a total of 10 drops of NaOH. Record your observations.
3. a. If a precipitate remains after the addition of 10 drops of NaOH, mix the contents of the tube, centrifuge and check the supernatant for complete precipitation. Add 10 more drops of NaOH if the precipitation was not complete. Wash the precipitate twice with 10 drops of distilled water. Save the precipitate for either Test IV or Test V.
   b. If no precipitate forms, dispose of these solutions in the proper waste container.
Test IV. NH₃ Test
1. IN THE HOOD, add 10 drops of 15 M NH₃ to the test tubes containing precipitate from Test III. Make sure to replace the cap on the ammonia reagent bottle after use.
2. Mix 2 minutes and centrifuge. Record your observations. If the ion did not give a precipitate in Test III, put an X in the corresponding box.

Test V. KSCN Test
1. Repeat Test III for each ion that resulted in a precipitate. Separate and wash the precipitate well.
2. Add 6 drops of 6 M HCl to the precipitate and mix.
3. Add 2 drops of KSCN solution and mix. Record your observations. If the ion did not give a precipitate in Test I, put an X in the corresponding box.

Test VI. Dimethylglyoxime (DMG) Test
1. Clean your test tubes. Add 10 drops of each individual metal solution (Pb²⁺, Ag⁺, Fe³⁺, Ni²⁺, Al³⁺) into its own test tube. Label the test tubes.
2. Test the pH of each solution with litmus paper.
3. If the solution is acidic, add 3 M NaOH dropwise until the solution is basic.
4. Once each solution is basic, add 2-4 drops of DMG solution to each test tube and mix. Record your observations.

Clean Up:
1. Dispose of all waste solutions in the proper waste containers.
2. Clean your test tubes and place them inverted on the test tube rack to drain.
3. Clean all glassware. Return all equipment to their original locations.
4. Check that you have a complete set of reagents and organize the reagents.

Data Analysis
Using your results from Part A and your answers to questions 1-6, devise a qualitative analysis scheme (flow chart) for analyzing a solution containing all five ions, Pb²⁺, Ag⁺, Fe³⁺, Ni²⁺, and Al³⁺. Questions to think about while constructing your flow chart include:
- Does the sequence of the tests matter?
- Which test should be done first?
- How can I separate the ions?
- How can I determine the presence or absence of a particular ion?
- Can I unambiguously identify the presence of each of the five ions in the solution? (The confirming test for the Al³⁺ ion may be a bit challenging.)

Draw your initial flow chart on the appropriate page of the Data Analysis Section. Prior to commencing further work in the laboratory, discuss your flow chart with your instructor.
Part B. Qualitative Analysis Scheme and Qualitative Tests for Five Ion Solution

After your scheme has been approved, test it by analyzing the “Five Known Ion” solution. Based upon your experimental results, make revisions to your scheme, if necessary. Include your improved flow chart on the appropriate page of the Data Analysis Section and show your final version to your instructor.

Part C. Analysis of an Unknown Solution

Once your instructor has approved your final flow chart, obtain an unknown solution and record its number in the Data Section. Use your flow chart to help you analyze your unknown solution. Determine which ions are present in your unknown by conducting chemical tests and providing experimental support for your determination. Record your lab procedure including your experimental results and observations on the appropriate page in the Data Analysis Section.

Written Lab Summary:

After completing this experiment, summarize your results in a short abstract or executive summary. This type-written summary must be a concise, organized, and readable description of your lab results which address the following areas:

1. Purpose
   What problem are you trying to solve in this experiment?
2. Experimental Methods
   Provide a brief overview of the lab protocols you used. Summarize your flow chart.
3. Results
   Briefly summarize your experimental results.
   How did you solve your problem?
   What ions were present in your unknown?
   Explain how you reached your conclusions.
   Your experimental results should support your claims.
4. Discussion/Conclusion
   What sources of error exist in this experiment?
   Why is qualitative analysis important?

Limit your summary to one or two type-written pages. Additional information on writing abstracts can be found at https://owl.english.purdue.edu/owl/resource/656/01/.