Introduction to Qualitative Analysis

Purpose

The purpose of this experiment is to (1) perform a series of control experiments for the interaction of silver, lead (II), nickel, barium, and iron (III) ions with various reagents, and (2) create a flow chart illustrating the confirmatory tests for the analysis of an unknown sample containing one or more of the above listed cations.

Introduction

Qualitative Analysis is a process used to identify whether certain substances are present in a solid or solution. When performing qualitative analysis, the experiment deals only with whether a substance is present or not. On the other hand, quantitative analysis is a determination of the *quantitity* of the substance present. Qualitative analysis is done by determining the presence (or absence) of a substance based on how the ions react to different reagents. A reagent is a solution that has the potential to react with one or more ions in a solution, often producing visible evidence of a chemical reaction. The following indications of a chemical reaction are commonly seen upon the addition of reagents:

1. The formation of a precipitate
2. A change in color of the solution and/or texture of a solution
3. Re-dissolving of a precipitate through the addition of heat
4. Evolution of a gas

When a reagent interacts with a solution and a precipitate is formed, it is suspended in a liquid known as the supernatant. The supernatant may be further analyzed by using a centrifuge. A centrifuge is a piece of lab equipment that spins a solution at a high speed for a set amount of time. As the solution spins, any solid or particulates will move down to the bottom of the test tube and compact. Once the sample is removed from the centrifuge, the supernatant can be separated from the precipitate by carefully pouring the solution into another test tube for analysis, leaving the precipitate in the original test tube. Centrifuges are also useful when differentiating between a color change in a solution and the formation of a precipitate. By centrifuging a sample, any precipitate will separate from the supernatant, giving a clear indication of whether a color change is due to a precipitate formation or a change in color of the solution.

Other laboratory techniques are often used during qualitative analysis. When a precipitate is formed, it is important to ensure that complete precipitation has occurred of the ion of interest. In order to test this, it is often advantageous to centrifuge a sample that has formed a precipitate, and once the precipitate and supernatant are separated, add another drop or two of the test reagent. If addition precipitation occurs, the ion of interest is still present within the solution. The test reagent should be added until no further precipitation occurs before the supernatant is removed. In addition, heat may be applied to force one compound into solution, leaving a second as a precipitate. Using these differences in solubility can assist in separating two ions that react with similar reagents and cannot be separated any other way.

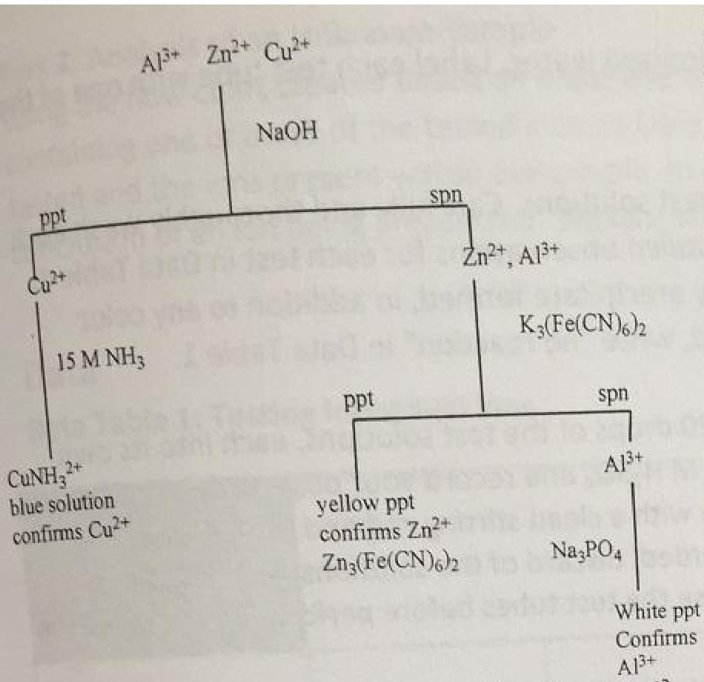

Figure 1: Flow chart for the analysis of Al^{+3}, Zn^{+2}, and Cu^{+2}

Qualitative analysis often involves performing several control tests with known samples in order to identify a positive result for ions of interest. The results of these control tests can be organized in a flow chart that identifies the steps required to produce a confirmatory result for the ion of interest. The flow chart can later assist for the rapid and comprehensive analysis of a solution which may contain multiple ions of interest. Consider the example flow chart seen in Figure 1, which demonstrates the analysis of aluminum, zinc, and copper (II) ions. The flow chart shows that when NaOH is added to the solution containing all, or some, of the ions, a blue precipitate forms if there are Cu^{2+} ions present. If the test tube is spun in the centrifuge, the ppt will be at the bottom of the test tube and the supernatant will contain the zinc and aluminum ions. Adding $K_3(Fe(CN)_6)_2$ solution to the supernatant causes zinc ions to form a yellow ppt. Adding the Na_3PO_4 reagent to the supernatant causes a white ppt to form, which confirms the presence of aluminum ions. Each flow chart created will be different based on the ions tested for.

Pre-Lab Questions

Read the Appendix entitled "Operation of a Centrifuge" and answer the following questions.

1. Why is it important to balance the centrifuge before using it?
2. How do you properly balance a centrifuge if an odd number of samples must be centrifuged?
3. Why is it important NOT to stop the centrifuge abruptly?
4. Given the following reaction:

$$Hg(NO_3)_2\ (aq) + 2HCl\ (aq) \rightarrow Hg_2Cl_2\ (s) + 2HNO_3\ (aq)$$

   Which compound would be present as a precipitate, and which one would be present in the supernatant?

Procedure

During this experiment, the following safety measures should be taken:

1. Be careful to avoid getting chemicals on yourself or others. If any spills occur, wash your hands immediately and notify your TA.
2. Acids and bases can cause burns on the skin and damage clothes. Be careful when handling these substances.
3. The silver solution will stain clothes and skin – avoid getting the solution on your hands or clothes. If, however, some gets on your skin, the dark stain will fade and disappear in a few days.
4. Be careful when handling hot glassware – use clamps or hot gloves.

Due to limited glassware present in the lab, be sure to wash your glassware between each experiment. Dirty glassware will contaminate solutions and potentially cause false results. Clean all glassware with soap and water, and rinse with deionized water before using again.

Part 1: Testing Individual Ions
Carefully wash five test tubes and rinse well with deionized water. Label each test tube with one of the five ions to be tested (Ag^{+}, Pb^{+2}, Ni^{+2}, Ba^{+2}, and Fe^{+3})

Perform the following chemical test on EACH of the test solutions. Carefully and thoroughly wash each test tube before performing the next test. Record *detailed* observations for each test in Data Table 1. Pay particular attention to formation and color of any precipitate formed, in addition to any color changes in the test solution. If no reaction is observed, write "no reaction" in Data Table 1.

1. **Addition of SO_4:** Use dropper bottles to add 20 drops of the test solutions, each into its own separate labeled test tube. Add 10 drops of 3 M H_2SO_4 and record your observations in Data Table 1. If a precipitate forms, mix the sample with a clean stirring rod and centrifuge the sample. Once all observations have been recorded, discard of the solutions and precipitate in the inorganic waste. Thoroughly clean and rinse the test tubes before performing the next step.

2. **Addition of HCl:** Add 20 drops of the test solution to the labeled test tubes. Add 10 drops of 6 M HCl. If a precipitate forms, mix the sample with a clean stirring rod and centrifuge the sample. Discard the supernatant. Wash the precipitate by adding 1 mL of deionized water to the precipitate and stir. Centrifuge the sample and remove the supernatant. Wash the precipitant a second time with 1 mL deionized water, discard the supernatant, and use the precipitate for Test 3.

3. **Heating of Precipitate and Addition of K_2CrO_4:** To the precipitate formed in Test 2, add 4 mL of deionized water and mix the solutions. Place the test tubes containing the precipitate in a hot water bath for 5 minutes and stir the mixture frequently. *Be sure not to contaminate solutions by using the same stir rod on multiple solutions!* Centrifuge the test tubes and carefully pour off the supernatant from each sample into a clean test tube. To the hot liquid, add 2 drops of K_2CrO_4. Record any observations in Data Table 1. Once all observations have been recorded, discard of any supernatants and precipitates in the inorganic waste. Thoroughly clean and rinse the test tubes before performing Test 4.

4. **Addition of NaOH:** Add 20 drops of the test solutions to the clean test tubes. To each test tube, add 4 drops of 6 M NaOH. If a precipitate forms, mix the sample and centrifuge. Record any observations in Data Table 5. Once the test tubes have finished in the centrifuge, discard the supernatant and use the same washing procedures described in Test 2 to wash the precipitate twice with deionized water. Discard any supernatants and use the precipitate in Test 5.

5. **Addition of NH_3:** To the washed precipitates, add 20 drops of 15 M NH_3 to each test tube and mix the solutions before centrifuging the mixture. Record any observations in Data Table 1. After recording observations, add 3 mL of DMG (dimethylglyoxime). Stir the solutions and record your observations.

Based on the results obtained from the known control samples, create a flow chart that allows for a rapid and comprehensive analysis of the five ions tested to be included in the Data section of the lab report. Use the example provided in the introduction (Figure 1) for assistance.

Part 2: Analysis of an Unknown Sample

Using the flow chart created based on experimental data from Part 1, analyze an unknown sample containing one or more of the tested ions. In Data Table 2, record the unknown number of the sample tested and the ions present within the sample. In addition, record any observations that lead to the conclusion of an ion being present/not present within the unknown sample.