Qualitative Analysis of Soluble Ionic Compounds

In this two-week experiment you will use a series of chemical tests to develop a qualitative analysis scheme (week one) that you will then use to identify the cation and anion in three soluble ionic compounds (week two). The possible identities of the cations and anions are shown here:

- cations: Cu^{2+}, Na^{+}, Ni^{2+}, Ba^{2+}, K^{+}, NH_4^{+}
- anions: CrO_4^{2-}, CO_3^{2-}, SO_4^{2-}, Cl^{-}, I^{-}

Each unknown is a soluble combination of one cation and one anion; your goal is to determine the identity of your three unknowns. Although you will complete the experiment individually, you will work with others as you develop your qualitative analysis schemes.

> ***Caution: Some of these cations and anions are harmful to the environment and/or toxic if ingested in high doses. Dispose of all waste in the appropriate waste bottle.***

Pre-lab Assignment

Read through the procedure for the first week before the first lab session. Come prepared with any questions you wish to discuss.

Week One: Analysis Of Known Salts

Your goal for this week is to explore a variety of chemical reactions that you can use to develop a qualitative analysis scheme that will allow you to identify each of the six cations and the five anions listed above. You will do this by beginning with solid samples of the nitrate salts of the cations (such as NH_4NO_3), and sodium salts of the anions (such as NaI).

Before you begin, make note of each compound's color. Cations often have distinctive colors—many transition metal ions have distinctive colors while main group and NH_4^{+} cations are typically colorless—and some anions have distinctive colors as well. Be cautious with this, however, as an ion's color may depend on its counter ion and on its crystallinity as well.

Testing the Cations

The following tests will help you distinguish between the six cations. As you perform a chemical test on a particular cation, record your observations in your lab notebook. Does the solution change color? Do you see evidence that a gas was released? Did a precipitate form? Did something else happen? If you observe a reaction, try to write a chemical equation that explains what happened and identify the type of reaction. If you do not observe a reaction, then simply record `NR` to indicate "no reaction."

Prepare a Stock Solution for Each Cation

Place a spatula tipful of a cation's nitrate salt in a test-tube and dissolve in approximately 5 mL of water. Stir the solution to dissolve the salt; if it does not dissolve completely, the set it aside and work on the other cations. When you return to this cation, if some of the solid remains undissolved, then centrifuge and transfer the overlying solution to a clean test-tube, leaving behind the undissolved solid. Make note of the solution's color. Label your test-tube. Repeat for each of the cations.

Chemical Tests

Working with one cation at a time, complete the following set of steps, paying particular attention to the directions as you will not complete all steps for each cation.

- **Step A**. Divide one of your cation solutions into two test-tubes. Cautiously (1–2 drops at a time) add 6M NaOH to one of these test-tubes. Mix the solution after each addition of NaOH and test the solution's pH using litmus paper. When the solution is just basic (turns red litmus paper blue), add an additional 3 drops of NaOH. If a precipitate forms, proceed to step A3. If no precipitate forms, proceed to step A1.
  - **Step A1**. If a precipitate did not form in Step A, transfer the contents of the test-tube into a 50-mL beaker. ***Working in a hood***, moisten a strip of red litmus paper with deionized water and place it on the convex side of a watch glass. Place the watch glass on top of the beaker such that the side with the litmus paper is facing down. If the litmus paper has not changed after 2-3 minutes, then heat the beaker gently on a hot plate. If the litmus paper turns blue, record your observation and proceed to the next cation; if the litmus paper does not change color, then proceed to step A2.
  - **Step A2**. If the litmus paper in step A1 did not change color, then complete a flame test on the portion of your cation solution to which NaOH was not added. Clean a test wire by alternating between dipping it in 6M HCl and heating it in the Bunsen burner's flame. When the wire no longer imparts a color to the flame, dip it in the unknown solution so that a thin layer of solution is suspended within the wire loop. Bring the edge of the test wire's loop to the edge of the flame's outer cone at a height equal to that of the flame's inner cone. Record the color of the flame and then proceed to the next cation.
  - **Step A3**. To distinguish between the several cations that produce a precipitate with NaOH, first centrifuge the contents of the test-tube to separate the precipitate from the overlying solution, which is called the supernatant. Remove the supernatant and discard it as waste. ***Working in a hood***, add 5 mL of 6 M NH_3 to the solid and mix thoroughly. Observe the contents of your test-tube and then follow the appropriate step from this set of three choices while continuing to work in a hood:
    - **Step A3a**. If the precipitate dissolves to produce a deep royal blue solution, then transfer the solution to a small beaker and add 3M $HC_2H_3O_2$ dropwise until the solution has a light blue color with no hint of purple. Add a few grains of NaCl. Gently sand an iron nail to expose a fresh surface and place it in the solution, undisturbed for 15–30 min while you begin work with another cation or begin work on the anions. Be sure to return and observe and record the result.
    - **Step A3b**. If the precipitate dissolves to produce a light bluish-purple solution, then transfer the solution to a beaker and add 3M $HC_2H_3O_2$ dropwise until the solution is nearly colorless. Add a few grains of dimethylglyoxime (dmg). Observe and record the result.
    - **Step A3c**. If the precipitate did not dissolve in ammonia, dissolve it in the same test tube using 6M HCl. Add 3 drops of 6M H_2SO_4. Observe and record the result.

Testing the Anions

The following tests will help you distinguish between the six cations. As you perform a chemical test on a particular cation, record your observations in your lab notebook. Does the solution change color? Do you see evidence that a gas was released? Did a precipitate form? Did something else happen? If you observe a reaction, try to write a chemical equation that explains what happened and identify the type of reaction. If you do not observe a reaction, then simply record `NR` to indicate "no reaction."

Prepare a Stock Solution for Each Anion

Place a spatula tipful of a anion's sodium salt in a test-tube and dissolve in approximately 5 mL of water. Stir the solution to dissolve the salt; if it does not dissolve completely, then centrifuge and transfer the solution to a clean test-tube, leaving behind the undissolved solid. Make note of the solution's color. Label your test-tube. Repeat for each of the anions.

Chemical Tests

Working with one anion at a time, divide the solution between three test-tubes and complete the following set of steps, paying particular attention to the directions as you will not complete all steps for each anion.

- **Step B1**. Cautiously (1–2 drops at a time) add 6M HNO_3 to the **first** of your three test-tubes. Mix the solution after each addition of HNO_3 and test the solution's pH using litmus paper, stopping the addition of HNO_3 when the solution is just acidic (turns blue litmus paper red). If your solution turns orange or bubbles or fizzes, record the result and proceed to the next anion; if not, then proceed to Step B2.
- **Step B2**. Add 10 drops of $BaCl_2$ to the **second** of your three test-tubes. If a white precipitate forms, centrifuge the contents of the test-tube to separate the precipitate from the overlying solution. Remove the supernatant and discard it as waste. Add several drops of 6M HNO_3 and record your observation. If a precipitate did not form when your first added $BaCl_2$, then proceed to Step B3.
- **Step B3**. Add 10 drops of 0.05 M $AgNO_3$ to the **third** of your three test-tubes. If a precipitate forms, record its color. Centrifuge the contents of the test-tube to separate the precipitate from the overlying solution. Remove the supernatant and discard it as waste. Add 1 mL of 6 M NH_3 to the precipitate while stirring. Record the results.

Write up for Week 1

Look back over the results of your reactions and develop a procedure that you can use to identify the cation and the anion in a soluble ionic compound whose composition is not known. You may choose to write out your procedure in the form of a series of steps, or you may choose to develop a flow chart.

Week 2: Analysis Of Unknown Salts

During this week you will work to identify three different unknown soluble ionic compounds using the scheme you developed last week. The unknown compounds have identification; be sure to record these numbers in your notebook, as well as all of your work.

Qualitative Analysis of Cations and Anions in your Unknown

Using the procedure you developed in week 1, analyze each unknown to identify its cation and anion. To make up a solution of an unknown, transfer the solid unknown to a small beaker and dissolve it in approximately 10 mL of water. Equally divide the solution between five test-tubes. Use two of these test-tubes to test for cations and the remaining three test-tube to test for anions.

Write up for Week 2

Turn in a neatly written report (use a word processor if, as is true for me, your handwriting is not neat) that addresses the following points for each unknown:

- Report your unknown's ID number, its chemical formula, and its name; for example, you might report it as 13A: $CuCl_2$ or copper(II) chloride.
- Report the color of the unknown as a solid and its color in solution, and indicate if this provided you with any useful information about the identity of the cation and/or the anion.
- Explain how you identified your unknown by documenting the specific tests you used and explaining how they support your conclusion. How confident are you about the unknown's identity? If you are not sure of your unknown's identity, then explain why? What are the likely candidates? What possibilities can you eliminate? Note: it is better to propose several options if you are not sure, than to guess incorrectly without mentioning possible alternatives!
- Turn in a copy of your procedure or flow chart with your report.