Qualitative Analysis

PURPOSE

To develop a separation scheme and confirmatory tests for Ag^{1+}, ${Hg_2}^{2+}$ and Pb^{2+} cations, and to use it to identify the ions in a sample of unknown composition.

GOALS

- To explore solubilities and reactivities of different metal ions.
- To logically develop a scheme to separate and identify a mixture of ions based on observations.

INTRODUCTION

Qualitative analysis is the process by which components of mixtures are separated and identified. Unlike quantitative analysis, where the amount of a particular material is measured, a qualitative analysis scheme simply *confirms* the presence or absence of certain materials. A common analysis is the identification of aqueous ions. In such an analysis, there are two distinct phases. First, a scheme must be developed to *separate* the ions from each other. Then, a different test is performed to uniquely *confirm* the identity of each separated ion. In this lab, we develop a scheme to separate and identify the silver ion (Ag^{1+}), the mercury(I) ion (${Hg_2}^{2+}$), and the lead(II) ion (Pb^{2+}). Mercury(I) ions exist as dimers in aqueous solution, and function as a +2 ion.

Ions are often separated in solution by their different solubilities. Although most chloride salts are soluble, some ions form insoluble compounds with chloride. Among the "insoluble" compounds, some dissolve in hot water. Some insoluble compounds can be brought into solution in the presence of ammonia, forming "metal ammine complexes" of the general formula ${M(NH_3)_x}^{y+}$ where x and y are specific to different metals. These three solubility characteristics (solubility of chloride salt, solubility of chloride salt in hot water, solubility of chloride salt in ammonia solution) form the basis of the separation scheme for Ag^{1+}, ${Hg_2}^{2+}$, and Pb^{2+}.

If one of the metal ions remains in solution after the addition of HCl and the others form insoluble solids **(precipitates)**, the addition of HCl effectively separates the soluble ion from the others. The soluble ion is separated by collecting the solid at the bottom of a test tube in a **centrifuge**, a device that creates a centrifugal force by rotation. After the precipitate is compacted, the **supernatant** (the liquid solution above the solid) is **decanted** (carefully poured off) into a separate container. The soluble ion is in the liquid supernatant while the insoluble ion is in the solid precipitate. Thus they are physically separated from each other when the liquid is poured off, leaving the solid behind. The solid precipitate is typically washed with water to help remove any traces of the soluble ions that remain. This prevents "false positive" test results later on.

In order to devise a scheme for separating a mixture of the cations in this lab, each ion will be tested individually for its solubility characteristics. You must carefully observe the behavior of each ion under the test conditions described in the procedure. The presence or absence of a solid and the accompanying colors of solids and solutions will provide important clues both for your separation scheme and the eventual identification of the ions. Deviation from the procedure may give different results, so be consistent in your technique.

When you think you have a particular ion isolated in solution, how do you prove it? One way is to do a chemical test on the solution and show that the results of that test are identical to those of a genuine sample of the ion. This type of confirmatory test forms the basis for identifying the ions you think you have separated. A true confirmatory test is unique to a particular ion. In other words, the presence of other ions will not give a false positive result. For the three cations in this experiment, the confirmatory test will be based on the addition of iodide ions. You will observe the behavior of the three cations in the presence of iodide, and then apply this test whenever you think you have isolated one of the three cations. The results of the test will confirm presence or absence of that ion.

In Part A, you will run qualitative, confirmatory tests on the individual ions to determine their behavior in the presence of iodide ions. In Part B, you will run qualitative tests on the individual ions to determine their solubilities in the presence of HCl, hot water and NH_3. In Part C, you will use your results to develop a scheme to separate and identify the ions in a mixture where all three ions are together. In Part D, you will use your scheme to separate and identify the cations in an unknown mixture. Completion of all four parts of this lab during the allotted lab period is difficult for one student pair. It is recommended that one student pair complete portions A and B; another student pair should complete portions C and D. The student groups can then exchange observations.

EQUIPMENT

6 small test tubes

1 test tube rack

1 test tube brush

3 glass stirring rods

1 250 mL beaker for waste collection

1 100 mL beaker for hot water bath with wire insert

1 pair of tongs

1 centrifuge

1 ceramic spot plate

1 hot plate

1 deionized water squirt bottle

REAGENTS

0.1 M $AgNO_3$

0.1 M $Hg_2(NO_3)_2$

0.1 M $Pb(NO_3)_2$

3 M HCl

3 M NH_3

0.2 M KI

deionized water

unknown solutions

SAFETY

HCl and NH_3 are corrosive. They can attack the skin and cause permanent damage to the eyes. If either solution splashes into your eyes, use the eyewash immediately. Hold your eyes open and flush with water. If contact with skin or clothing occurs, flush the affected area with water. Have your lab partner notify your instructor about the spill. The solutions also have irritating vapors. They should be used in a fume hood; avoid inhaling the vapors.

Lead and mercury are toxic if ingested. You will be working with very small quantities of these materials, so the hazard is minimal. Nonetheless, be aware of small splashes and spills. Clean them up immediately. Wash your hands if you think you have spilled solution on them, and certainly before leaving lab. Avoid putting anything in your mouth while in lab, ex. chewing on fingernails, pens and pencils.

Silver solution will form dark spots on skin if spilled. The spots will not appear for about 24 hours, as the ions are slowly reduced to the metal. They are not hazardous, and will fade in a few days.

WASTE DISPOSAL

All solutions used or produced in this experiment must go into the *waste container*, as they all potentially contain heavy metal cations. Label a 250 mL beaker as a waste container for use at your bench. Empty it into the lab waste container when you have finished your work, then rinse it with a few mL of water. These rinsings should also go into the lab waste container. Afterwards, the test tubes can be washed with soap and water in the sink. The solids should also go into the waste container; **nothing should go into the sink.**

PRIOR TO CLASS

Please read the following section of the Introductory Material:

- Centrifuge[1]

LAB PROCEDURE

Please print the worksheet for this lab. You will need this sheet to record your data.

Determine among four students which pair will perform Parts A and B, and which pair will perform Parts C and D.

Part A: Confirmatory Tests for Individual Ions

[1] ../equipment/manual.html#centrifuge

In a ceramic well plate, put three drops of each metal solution, one solution per well. Add 2 drops of 0.2 M KI to each. Record your observations in Data Table A for the well plate solution (solid? color? etc.). The remainder of the entries in Data Table A will be completed during Part B of the experiment.

Part B: Tests with Individual Ions.

Solubility in HCl

1. Place approximately 75 mL of tap water in a 100 mL beaker. Place the wire insert in the beaker. Place the beaker on the hot plate and heat the water to a gentle boil.

2. Label three small, clean test tubes with the symbols of the cations in this experiment, Ag^{1+}, $Hg_2{}^{2+}$ or Pb^{2+}. Place 5 drops of the cation solution in the appropriately labeled tube.

3. Place a small, clean stirring rod in each tube. Add three drops of 3 M HCl to each solution and stir. Record your observations in Data Table B (solid? color? etc.).

4. Perform a confirmatory test for any solution in step 3 for which a solid does **not** form. Confirmatory Test: Add 5 drops of 0.2 M KI to the test tube. Enter your observations in Data Table A. The contents of this test tube may be discarded into the waste.

If a solid does form in step 3, do **not** perform a confirmatory test. Enter N/A in Data Table A for the solution from HCl and continue to the next step.

5. In cases where a precipitate formed in step 3, isolate the solid by centrifuging and decanting the supernatant liquid. The supernatant may be discarded into the waste in this test. Wash the precipitate (to remove any remaining HCl) by adding 10 drops of deionized water and mixing with a stirring rod. Be sure to break up any clumps of solid that are present. Centrifuge the sample and decant the supernatant liquid. This "washing liquid" may be discarded into the waste.

Solubility in Hot Water

6. Add 10 drops of deionized water to the washed solids from step 5 and place the tubes in the boiling water bath for several minutes, stirring frequently with a clean stirring rod. Record your observations in Data Table B (color change? redissolve? etc.).

7. Perform a confirmatory test for any solution in step 6 for which solid dissolves. Confirmatory Test: Add 5 drops of 0.2 M KI to the test tube. Enter your observations in Data Table A. The contents of this test tube may be discarded into the waste.

If a solid does remain in step 6, do **not** perform a confirmatory test. Enter N/A in Data Table A for the solution from hot water and continue to the next step.

8. For the samples from step 6 where a precipitate remains, isolate the solid by centrifuging and decanting the supernatant liquid. The supernatant may be discarded into the waste in this test.

Solubility in Ammonia

9. Add 10 drops of 3 M NH_3 to the solids that did not dissolve in hot water in step 6. Record your observations in Data Table B (color change? redissolve? etc.).

10. Perform a confirmatory test for any solution in step 9 for which solid dissolves. Confirmatory Test: Add 5 drops of 0.2 M KI to the test tube. Enter your observations in Data Table A. The contents of this test tube may be discarded into the waste.

Note: One partner should perform Part C while the other partner performs Part D simultaneously. This will allow comparison between a solution that is *known* to contain Ag^{1+}, Pb^{2+} and $Hg_2{}^{2+}$ (Part C) with one that may or may not contain all three (Part D). Be very careful with data collection since both partners will be performing the same tests on different solutions.

Part C: Developing a Separation Scheme for a Mixture of All Three Ions

Add 5 drops of *each* cation solution (Ag^{1+}, Pb^{2+} and $Hg_2{}^{2+}$) to a single test tube. This will simulate conditions in an unknown mixture.

Solubility in HCl

1. Add a few drops of 3 M HCl. A precipitate will appear. Continue adding HCl until no more solid appears to be forming. Centrifuge the sample.

2. Add another drop of HCl. If no precipitate forms, go to step 3. If more solid appears, add HCl until no more forms. Centrifuge again, and add another drop of HCl. Continue this until no more solid forms, then go to step 3. Record your observations in Data Table C1.

3. Decant the supernatant liquid into a clean test tube and perform a confirmatory test. Confirmatory Test: Add 5 drops of 0.2 M KI to the test tube. Record your observations in Data Table C2. The contents of this test tube may be discarded into the waste.

4. Wash the solid with deionized water as you did in step 5 of Part B. Decant the washing liquid into the waste. Washing the precipitate removes any soluble ions and prevents problems in future steps, such as false positives for confirmatory tests.

Solubility in Hot Water

5. Add 10 drops of deionized water to the precipitate that formed in step 1 and was washed in step 4. Place the tube in a boiling water bath for several minutes, stirring frequently with a clean stirring rod. Record your observations in Data Table C1 (color change?, redissolve?, etc.)

6. Centrifuge your sample. Decant the supernatant liquid into a clean test tube and perform a confirmatory test. Confirmatory Test: Add 5 drops of 0.2 M KI to the test tube. Record your observations in Data Table C2. The contents of this test tube may be discarded into the waste.

7. Wash the solid as you did in step 5 of Part B. Discard the washing liquid into the waste.

Solubility in Ammonia

8. Add 10 drops of 3 M NH_3 to the solids that did not dissolve in hot water and were washed in step 7. Record your observations in Data Table C1.

9. If a solid remains, centrifuge your sample. Decant the supernatant liquid into a clean test tube and perform a confirmatory test. Confirmatory Test: Add 5 drops of 0.2 M KI to the test tube.

Record your observations in Data Table C2. The contents of this test tube may be discarded into the waste.

Part D: Separating and Identifying Components of an Unknown Mixture.

1. Place 15 drops of an unknown mixture in a clean test tube. Record the unknown code in the heading of Data Table D1. The unknown contains some combination of Ag^{1+}, Pb^{2+} and ${Hg_2}^{2+}$ ions.

2. Follow steps 1 - 9 from Part C on the unknown sample while your partner performs these steps on the known mixture. At each step, compare the unknown results to the known results to identify the ions in the unknown mixture. At each step, remember to:

- Record your observations in Data Tables D1 and D2.
- **Save** all supernatant solutions for confirmatory tests.
- Wash solid samples before going on to avoid contamination with soluble ions.

3. Turn off your hot plate. When cooled, pour your water bath out into the sink. Use the tongs provided to remove the beaker if it is still warm.

4. After answering the questions, collect all your solutions in your waste beaker. Rinse all glassware into your waste container with a minimum amount of deionized water. Deposit the contents of your waste beaker into the waste container on the side shelf. Rinse with a minimum amount of deionized water.

5. Wash and dry all your equipment and return it to the set-up area where you found it.

6. Before leaving, go to a computer in the laboratory and enter your results in the In-Lab assignment. If all results are scored as correct, log out. If not all results are correct, try to find the error or consult with your lab instructor. When all results are correct, note them and log out of WebAssign. The In-Lab assignment must be completed by the end of the lab period. If additional time is required, please consult with your lab instructor.

