Objectives

- Use qualitative analysis to analyze an unknown

Background

Qualitative Analysis

Qualitative analysis is the process of deciding what is in a particular sample. The process determining the amount present is called quantitative analysis. The processes are obviously not completely disconnected because a thorough knowledge of the contents of a sample is often required before quantitative analysis can be performed. In some case, the same reactions that are used to detect the presence of an unknown can be used to determine the amount present. In order to make this exercise manageable, we will focus our attention on only five anions Cl^-, I^-, CO_3^{2-}, PO_4^{3-}, and SO_4^{2-}. The reactions of these ions are important in all phases of chemistry and biochemistry so a solid background will be of benefit in many of your future studies. It will be obvious in the subsequent material that the chemistry of anions cannot be completely separated from the chemistry of the cations. Traditionally, the chemistry of chloride and iodide are developed through an examination of the reactions with silver ion. For example:

$$Ag^+_{(aq)} + Cl^-_{(aq)} \rightarrow AgCl_{(s)}$$

In practice the above reactions usually are performed by adding a solution containing the anion to a solution containing silver ion. The silver ion is obtained by dissolving a soluble silver salt, like silver nitrate, in distilled water (tap water has enough chloride to cause a reaction). The word “solid” in parentheses indicate that a solid material is formed that falls out or “precipitates” from solution. In some cases, the precipitate immediately settles to the bottom of the reaction container. In other cases, the solution becomes cloudy. We will leave the reactions of other anions and cations for you to discover. Subsequent reactions of the precipitates can be used for further analysis. For example, we can test to see what reagents will dissolve the precipitates. We can illustrate the process by the reaction of AgCl with NH_3.

$$AgCl_{(s)} + 2NH_{3(aq)} \rightarrow Ag(NH_3)^{2+} + Cl^-$$

The AgCl dissolves by reaction with ammonia to form a new chemical species. Again we will leave the remaining reactions for you to discover. In some case there are very specific reactions that can be used to identify an anion. For example, carbonate ion reacts with acid to generate CO_2 gas which is readily identified. Molybdate ion is another example. In this case there is a complex reaction with phosphate that is diagnostic for that phosphate ion. Again you will see these reactions as you proceed through the exercise. If all samples contained only a single anion the need for much of the chemistry described above would be eliminated. In reality, anions typically occur in mixtures and the tests must either be insensitive to the presence of other anions or we must separate these potential interferences before performing the test. In the last part of the exercise you will be given an unknown with anions. Your job will be to work out a procedure that allows unique identification of all of the anions in the sample.

Materials

- 0.5 M solutions of:
  - NaCl
  - KCl
  - Na_2CO_3
  - Na_3PO_4
  - Na_2SO_4
- 6M HNO_3
- 0.2M $AgNO_3$
- 3M NH_3(aq)
- 0.3M $Ba(NO_3)_2$
- 15M HNO_3
- 1M $(NH_4)(MoO_3)_4^+$
- Test tubes
- Litmus paper

Safety goggles are required!

Concentrated nitric acid is extremely corrosive and will cause serious burns; handle with extreme caution.

Procedure

Qualitative Analysis

Standardizing anion solutions

1. Obtain 1mL samples of 0.5M solutions of the salts NaCl, KI, Na_2CO_3, Na_3PO_4, and Na_2SO_4 in separate, clean, labeled test tubes. These solutions will be the source of known anions. For these small volumes, it is best to manipulate your samples using disposable pipettes. Using these pipettes as droppers, 1mL is approximately 20 drops.

2. Using deionized water, dilute each of the anion solution samples to a final volume of 5mL.

3. Each of the anion solutions should be subjected to each of four tests unless otherwise noted in the conditions of the test. All tests should be performed in a clean, dry test tube.

Test 1 (performed on all anion solutions)

4. Add drops of 6M HNO_3 to the anion solutions until they become acidic. The acidity of the solution will be detected using blue litmus paper. Use your stirring rod to touch a drop of the solution onto the litmus paper. Be sure to test each solution before addition of HNO_3. If the solution is already acidic, add only one drop of HNO_3.

5. Add 15 drops of 0.2M $AgNO_3$ to each solution. Note all observations in your data table.

6. If a precipitate forms, let it settle to the bottom of the test tube. After the solid has settled, decant the supernatant liquid. Save the precipitate for Test #2.

Test 2 (performed only on precipitates of Test 1)

7. If no precipitate formed in Test 1, draw an X in the appropriate box for Test 2. Test 2 is not performed on anions which did not form a precipitate in Test 1.

8. Wash each precipitate obtained from Test 1 using deionized water twice. Washing includes adding deionized water, stirring, letting settle, and then decanting the supernatant liquid.

9. Place a small amount of each precipitate in a clean, dry test tube and add 10 drops of deionized water and 10 drops of 3M NH_3 (aq) to each and mix for several minutes. Record your observations.

10. Let the solid settle to the bottom and then decant the supernatant liquid into another test tube. To this supernatant liquid, add 6M HNO_3 until the solution is acidic. Record your observations.

Test 3 (performed on all anion solutions)

11. Place 20 drops of each anion solution into clean, dry test tubes. Test each solution with red litmus paper to determine if the solution is basic. If the solution is basic, add 1 drop of 3M NH_3 (aq). If the solution is not basic, add NH_3 (aq) dropwise until the solution is basic.

12. Add 15 drops of 0.3M $Ba(NO_3)_2$ to each solution. Record your observations.

13. If a precipitate forms, let the solid settle to the bottom and decant off the supernatant liquid. Save this precipitate for Test 4.

Test 4 (performed on precipitates from Test 3)

14. If no precipitate formed in Test 3, draw an X in the appropriate box for Test 4. Test 4 is not performed on anions which did not form a precipitate in Test 3.

15. Wash each precipitate obtained from Test 3 using deionized water twice. Washing includes adding deionized water, stirring, letting settle, and then decanting the supernatant liquid.

16. Add a small amount of each precipitate to a clean, dry test tube and add 3mL of 15M HNO_3, then add 15 drops of deionized water to each and mix. Place the test tubes in a gently boiling water bath for approximately 5 minutes. Every 30 seconds, stir the mixture. Record your observations during the heating process.

17. To each test tube, add 5 drops of 1M ammonium molybdate $(NH_4)(MoO_3)_4^{+}$. Record your observations.

Determination of Unknown

18. Ask your lab instructor to assign you an unknown solution. Record your unknown solution number.
19. Devise a way to either determine or rule out the presence of each anion in solution.
20. Determine which, if any, anions are present in your solution. Record them in your data sheet.

Pre-Lab Questions

Prepare for lab by completing and understanding the answers to these questions. Refer to the Background or another resource, such as your textbook, if necessary.

1. Why would using these procedures not work for potassium nitrate, and what would you have to do differently to test for nitrates?

2. What would happen to your samples if you used tap water to make the dilutions required in this lab?

3. From question 2, if you accounted for an anion in the tap water could you be certain that you accounted for all of the anions in solution without testing the water? Explain your answer.

Lab 3: Qualitative Analysis Report Sheet

Name ____________________ Section ____________________

Date ____________________ Instructor ____________________

Qualitative Analysis

Test 1	Cl^-	I^-	SO_4^{2-}	PO_4^{3-}	CO_3^{2-}
6M HNO_3 + 0.2M $AgNO_3$	________	________	________	________	________

Test 2	Cl^-	I^-	SO_4^{2-}	PO_4^{3-}	CO_3^{2-}
3M NH_3	________	________	________	________	________
6M HNO_3	________	________	________	________	________

Test 3	Cl^-	I^-	SO_4^{2-}	PO_4^{3-}	CO_3^{2-}
3M NH_3 + 0.3M $Ba(NO_3)_2$	________	________	________	________	________

Test 4	Cl^-	I^-	SO_4^{2-}	PO_4^{3-}	CO_3^{2-}
15M HNO_3 + heat	________	________	________	________	________
Ammonium molybdate	________	________	________	________	________

Unknown # ____________________

Anions present ____________________

Post-Lab Questions

1. Describe the tests performed to determine if each anion was or was not present in your unknown solution.

2. Considering your knowledge of the reaction between chloride anions and silver cations, propose a procedure to quantify the concentration of chloride ion in a solution.

3. Describe a scenario in which this experiment could be extended to a real world application.