Separation and Qualitative Analysis of Anions

The second part of your qualitative analysis experiment is the testing of anions. The process of analysis will be similar to Experiment 14A. Thus, if you did not complete 14A, it will be helpful to read the introductory comments and review the procedure.

The primary difference between cation analysis and anion analysis is in the separation steps; with cations, separation is common, but with anions, separation is rare.

As with Experiment 14A, you will first prepare a solution containing anions of your choice (the “known” solution), and then you will test an unknown solution to identify the anions it contains.

OBJECTIVES

In this experiment, you will

- Prepare and analyze a solution that contains six selected anions.
- Analyze an unknown solution that contains a selection of anions.

MATERIALS

Test Solutions and Substances
- 6 M nitric acid, HNO₃
- 0.1 M silver nitrate, AgNO₃
- 6 M acetic acid, CH₃COOH
- 6 M ammonia, NH₃
- 6 M sodium hydroxide, NaOH
- 0.1 M iron (III) nitrate in 0.6 M HNO₃
- 0.1 potassium permanganate, KMnO₄
- 0.1 M barium chloride, BaCl₂
- barium hydroxide Ba(OH)₂
- granular aluminum
- mineral oil

Anion Solutions
- 0.2 M sodium chloride, NaCl
- 0.2 M potassium iodide, KI
- 0.2 M sodium carbonate, Na₂CO₃
- 0.2 M sodium bromide, NaBr
- 0.2 M sodium sulfate, Na₂SO₄
- 0.2 M sodium nitrate, NaNO₃

Safety Chemicals for Spills
- baking soda, NaHCO₃(s)
- vinegar (5% acetic acid solution)

Equipment
- nine test tubes, 13 ×100 mm
- test tube holder
- test tube rack
- corks to fit test tubes
- 400 mL beaker for rinsing stirring rods
- 250 mL beaker for hot water bath
- plastic Beral pipets, graduated is best
- several stirring rods
- ring stand, ring, wire gauze, burner or hot plate
- cotton
- pH paper or Vernier pH Sensor
- capillary pipet
**PRE-LAB EXERCISE**

1. Prepare a chart for recording your observations as you test for the presence of anions. A sample chart, with the test results for a known solution, is shown below.

<table>
<thead>
<tr>
<th>Test</th>
<th>Procedure</th>
<th>Known Solution</th>
<th>Unknown Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Add AgNO₃ to solution in Test Tube 1. Centrifuge. Pour the supernatant into Test Tube 2. Wash ppt.</td>
<td>White ppt forms. Other ions in the liquid in Test Tube 2.</td>
<td>Cl⁻ present.</td>
</tr>
</tbody>
</table>

2. Label your test tubes clearly, and arrange your chemical reagents in an orderly manner, so that you will be certain of the results of each test in the qual scheme.

3. Wash and rinse all of your glassware with distilled water. Tap water will contain some of the ions for which you are conducting the tests. If you use a glass stirring rod, make sure that it is rinsed between uses. You may store your glass stirrers in a 400 mL beaker that is about ¾ full with distilled water.

4. Prepare a hot-water bath in a 250 mL beaker, which will be used intermittently during the experiment.

5. You will be testing two solutions, one that you will prepare and one unknown solution. Decide, before starting this experiment, whether you will be testing the solutions simultaneously or not. There are advantages and disadvantages either way, and it depends largely on how comfortable you are with this procedure.

6. At certain points in the testing, you will be checking the pH of a solution. You may use conventional pH paper to check pH or you may use a Vernier pH Sensor. If you use a pH Sensor follow the steps below to set up the sensor.
   a. Connect a LabPro or CBL 2 interface to the computer or handheld with the proper cable.
   b. Connect the pH Sensor to Channel 1 of the interface.
   c. Start the data collection program and set up the program for a pH Sensor.
   d. pH readings will be displayed on the computer or handheld screen.

**SAFETY**

The acid and base solutions that you will use in this experiment have high molar concentrations. Even though you will use only a few drops of these solutions, they must be used very carefully. Use the normal precautions and processes for diluting and cleaning up any spills of the acids and bases. Locate the containers of vinegar and baking soda that have been placed in the lab for use in neutralizing spills.
PROCEDURE

Preparation of the Known Solution for Analysis
1. Obtain and wear goggles.
2. Set up nine test tubes in a test-tube rack. Label the test tubes 1-9. Prepare a hot water bath in a 250 mL beaker.
3. Prepare a known solution in Test Tube 1 by mixing 1 mL of each ion that you wish to test. You will have about 6 mL of solution.

Separation of the Halides and Confirmation of Chloride ions
4. Transfer 10 drops of the known solution to Test Tube 2. Check the pH of the solution. If it is not acidic, add 6 M HC₂H₃O₂ solution dropwise until the solution is acidic. Add 10 drops of 0.1 M AgNO₃ solution to Test Tube 2 and stir the mixture. A white precipitate will form, which is a mixture of one or more of the following compounds: AgCl, AgBr, and/or AgI. Centrifuge the test tube and pour off the supernatant liquid. Wash the precipitate with 0.5 mL of distilled water, centrifuge the test tube, and pour off the wash water.
5. Add 0.5 mL of NH₃ solution to the precipitate in Test Tube 2 and stir the mixture. Centrifuge the test tube and transfer the supernatant to Test Tube 3. Discard the precipitate in Test Tube 2 as directed, and clean the test tube.
6. Add 1 mL of 6 M HNO₃ solution to Test Tube 3. The solution will warm up and produce a bit of smoke from the acid-base reaction, regardless of the presence or absence of Cl⁻ ions. Check the pH of the mixture. If it is not acidic, add more 6 M HNO₃ dropwise until the liquid is acidic. If a white precipitate appears in the acidic liquid, then Cl⁻ ions are present.
7. Discard the contents of Test Tube 3 as directed, and clean the test tube.
8. Transfer 10 drops of the known solution (Test Tube 1) to Test Tube 4. Add 6 M HNO₃ solution until the solution is acidic. Add 1 mL of 0.1 M Fe(NO₃)₃ solution and stir the mixture. Add 1 mL of mineral oil, use a cork to stopper the test tube, and shake the test tube for 30 seconds. If the mineral oil layer is a pink-to-purple color, then there are I⁻ ions present.
9. Use a capillary eyedropper or a plastic Beral pipet to remove the mineral oil layer from Test Tube 4. Discard the oil layer as directed. Add another 1 mL of mineral oil to Test Tube 4, stopper, shake the test tube for thirty seconds, and discard the oil layer. Continue this step until the oil layer is clear, which ensures that you have separated all of the I⁻ ions from the mixture.
10. Add 0.1 M KMnO₄, to the liquid in Test Tube 4, dropwise with stirring until the solution remains a pink color. Add 1 mL of mineral oil, stopper the test tube, and shake it for thirty seconds. If the mineral oil layer is a brown color, then there are Br⁻ ions present. Discard the contents of Test Tube 4 as directed, and clean the test tube.

Status Check
At this point in your analysis, you will have tested for three anions: Cl⁻, I⁻, and Br⁻. Test Tube 1 contains your original solution, and all of the other test tubes should be empty.
Analyze the Solution for the Presence of CO$_3^{2-}$

11. Look back at your notes for the previous tests. If you observed bubbles forming when acid was added to the original solution, then CO$_3^{2-}$ ions are most likely present. To confirm the presence of CO$_3^{2-}$ ions, add 2 mL of saturated Ba(OH)$_2$ solution to Test Tube 5. Transfer 10 drops of the known solution (Test Tube 1) to Test Tube 6. Add 0.5 mL of 6 M HNO$_3$ solution to Test Tube 6, and place the test tube in a hot water bath.

12. If bubbles form in Test Tube 6, then quickly take a plastic Beral pipet, squeeze the bulb flat, place the tip of the pipet over Test Tube 6 (just above the liquid) and slowly release the bulb to capture some of the carbon dioxide that is being produced. If bubbles have not formed in Test Tube 6, then there is no CO$_3^{2-}$ present.

13. Place the tip of the Beral pipet of CO$_2$ gas into Test Tube 5, which contains Ba(OH)$_2$ solution. Slowly squeeze the bulb to transfer the gas. You should see a cloudy white precipitate form, confirming the presence of CO$_3^{2-}$ ions. Discard the contents of Test Tubes 5 and 6 as directed, and clean the test tubes.

Analyze the Solution for the Presence of SO$_4^{2-}$

14. Add 0.5 mL of the known solution (Test Tube 1) to Test Tube 7. Add 6 M HC$_2$H$_3$O$_2$ solution dropwise until the contents of Test Tube 7 are acidic. Add 0.5 mL of 0.1 M BaCl$_2$ solution. If a white precipitate forms, then there are SO$_4^{2-}$ ions present. Discard the contents of Test Tube 7 as directed and clean the test tube.

Analyze the Solution for the Presence of NO$_3^{-}$

15. Add 1 mL of the known solution (Test Tube 1) to Test Tube 8. Add 6 M NaOH solution dropwise until the contents of Test Tube 8 are basic, and then add 6 drops more. Use a plastic Beral pipet to carefully transfer the liquid in Test Tube 8 to Test Tube 9. Note: Do not splash the liquid and avoid getting any liquid along the side of the test tube.

16. Add a tiny amount of aluminum granules to Test Tube 9. Place a small cotton ball loosely about halfway down into the test tube, but not touching the liquid. This is done to prevent spattering. Hang a strip of litmus paper (or pH paper) in the test tube so that the tip of the paper is just above, but not touching, the ball of cotton.

17. Place Test Tube 9 in the hot-water bath. Leave the test tube in the bath until the liquid in the test tube starts to bubble strongly. Be sure that neither the solution nor the cotton ball touches the litmus paper. Remove the test tube from the bath and allow it to cool. A slow color change, within three to five minutes, of the litmus paper from pink to blue indicates the evolution of NH$_3$ gas from the mixture and confirms the presence of NO$_3^{-}$ ions. Discard the contents of Test Tubes 1, 8, and 9 as directed.
DATA ANALYSIS

Identify the anions that were present in your unknown solution. Describe, in detail, how you used the results of your testing to identify the contents of your unknown solution. As you prepare your concluding remarks, consider the following:

1. Discuss the similarities in the tests to confirm the presence of chloride ion and silver ion (from 14A).

2. Discuss the use of $K_{sp}$ and solubilities to separate AgCl from AgBr and AgI.

3. Use reduction-potential values to explain which halides can be oxidized by Fe$^{3+}$ or by MnO$_4^-$ in acidic solution.

4. Explain why the test for I$^-$ must be done before testing for Br$^-$. 

5. Write the half-reactions that describe the test that you conducted for NO$_3^-$. 

6. In Step 14, Ba$^{2+}$ was added to the original solution, which contained all six anions. The reaction produced the precipitate BaSO$_4$, but not BaCO$_3$. Earlier, in Steps 11-13, BaCO$_3$ forms as a precipitate to confirm the presence of carbonate ions. Why didn’t BaCO$_3$ precipitate in Step 14?