

# Experiment 4: The Qual Scheme

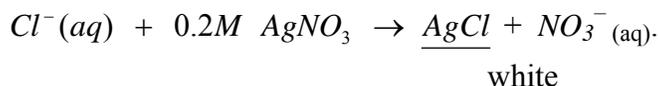
## Selected Anion Analysis

### Anion Elimination Tests

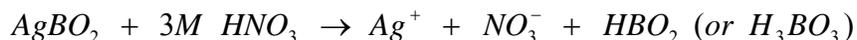
In this experiment you will analyze for the presence of five anions. The knowns are provided as sodium salts (all white crystals) and 0.2M solutions of the same salts. The anion unknown provided is a mixture of the solids ground together. While many anions are known by their intrinsic color, none of these five are colored. Nor will there be any keys to identification by knowing the cation since all sodium salts are soluble. Known samples are to be used separately in three experiments described below. In these experiments you may want to join with a partner.

### Experiment 1 (Essentially Procedure 22)

Using a few drops of each of the 0.2M solutions of anion knowns in separate labeled tubes, add 0.2M  $\text{AgNO}_3$ . Note which anions produce precipitates (all are insoluble silver salts) and note the color of each. For each anion write an equation of this form:



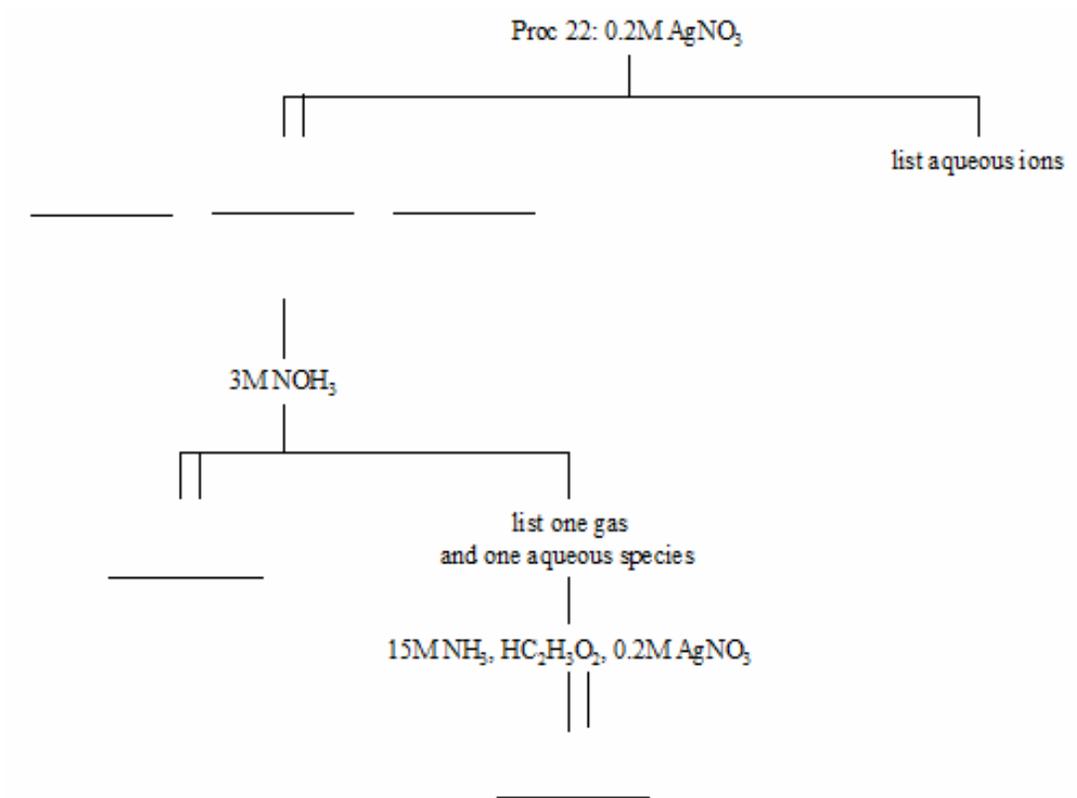
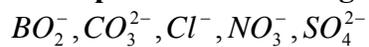
Where precipitates occurred, centrifuge the sample and discard the decantate. Treat the precipitates with 3M  $\text{HNO}_3$  (watch for bubbles) to find which dissolve. Write equations for those silver salts which dissolve in 3M  $\text{HNO}_3$  in this form:



Finally, for those silver salts which did dissolve in acid, make those solutions just basic with 15M  $\text{NH}_3$ , then just acidic with 5M  $\text{HC}_2\text{H}_3\text{O}_2$  and add two drops of 0.2M  $\text{AgNO}_3$ . Note which analyte anions form their silver salts.

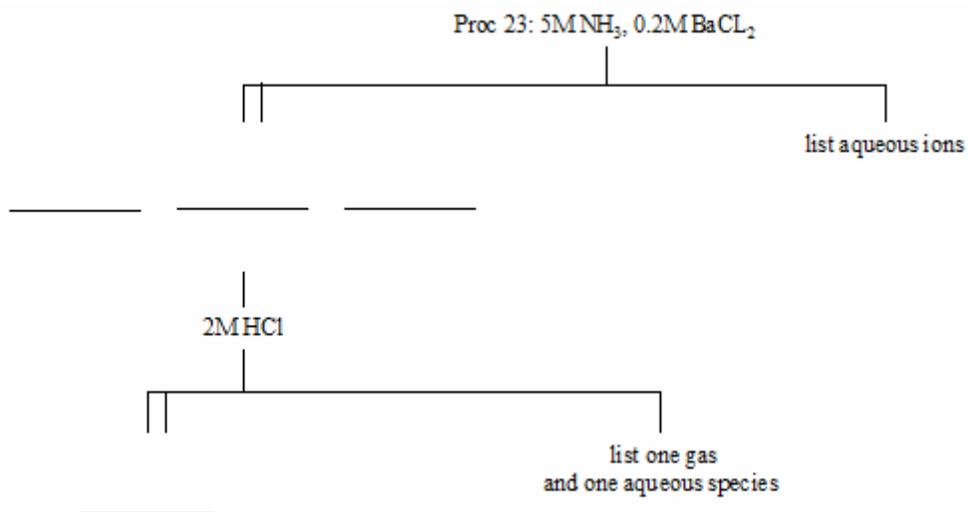
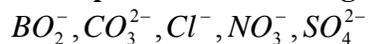
Fill out the flow diagram "Anion Experiment One" with the formulas of products and colors on the basis on your experimental results. Try to get this right because you'll need to consult it later. Because testing a basic solution with silver nitrate may cause the precipitation of  $\text{AgOH}$ , you should also do this experiment. Take one small drop of 8M  $\text{NaOH}$  and dilute it to 10mL in your 10-mL graduated cylinder, mix thoroughly. Take about one half mL of this, and add a drop of 0.2M  $\text{AgNO}_3$ . Observe the brown precipitate. When testing basic salts such as  $\text{Na}_2\text{CO}_3$ , this brown precipitate may surprise you.

### Anion Experiment One Diagram



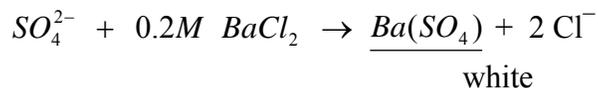
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### Anion Experiment Two Diagram

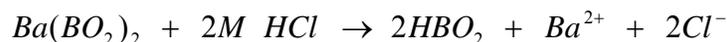


### **Experiment 2 (Essentially Procedure 23)**

Using two drops of each of the known solutions in separate labeled tubes, first add a drop of 5M NH<sub>3</sub> to each. Then add 2 or 3 drops of 0.2M BaCl<sub>2</sub>. Note which produce precipitates and their color. Write equations for precipitate formations in this form:



Where precipitates are formed, centrifuge and discard the decantates. Treat the precipitates with 2M HCl (look for bubbles). Write equations for precipitate dissolution in acid in this form:



Fill out the flow diagram “Anion Experiment Two” with the formula of products and colors on the basis of your experimental results.

### **Experiment 3 (Essentially Procedure 24)**

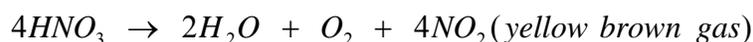
Take a small pinch of each of the five known sodium salts of the anions in five separate dry test tubes and treat each successively with 2 or 3 drops of 18M H<sub>2</sub>SO<sub>4</sub>. At this time let us remind you how corrosive is concentrated H<sub>2</sub>SO<sub>4</sub>. Treat it with great respect. Don't point the tube towards anyone, especially later when heating. And of course many acid gases are produced; smell them only by wafting. As you add the conc H<sub>2</sub>SO<sub>4</sub>, watch for effervescence (bubbles) and discoloration in the acid solution.

Two of the five salts produce gases. Carbonates produce the mildly acid, colorless, odorless gas CO<sub>2</sub>, with any strong acid solution. But the other will bubble only with very concentrated strong acids. Chlorides, on treatment with 18 M H<sub>2</sub>SO<sub>4</sub> produce HCl, a colorless sharply acidic gas which fumes in moist air.

Write two equations for the formation of these gases.



Then, with the three salts which did not produce gases, take each mixture and heat it carefully till hot and look for further activity. You will note that nitrate produces yet another yellow brown gas by decomposition of the nitric acid formed.



### ***Procedure 22***

When analyzing the “anion unknown,” remember that you must interpret the results in view of the fact that more than one anion is present. In the case of ‘single salt’ unknown consider whether any acid was used to put the salt in solution.

Place three drops of the solution to be analyzed in a test tube and add 2 drops of 0.2M AgNO<sub>3</sub>. Note the color of any precipitate. Centrifuge and test for complete precipitation with 0.2M AgNO<sub>3</sub>. Centrifuge and discard the decantate. Treat the precipitate with plenty of 3M HNO<sub>3</sub>, look for bubbles and any remaining precipitate. Centrifuge and treat the decantate with 15M NH<sub>3</sub> dropwise until just basic, then with 5M HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub> until just acid. Add another drop of 0.2M AgNO<sub>3</sub> and note the color of any precipitate. Under these conditions no problem with AgOH or Ag<sub>2</sub>O should occur.

### ***Procedure 23***

As with Proc 22 interpretation will depend on whether the sample has several or a single anion. To three drops of solution to be analyzed add 5M NH<sub>3</sub> until the sample is just basic, usually one drop if the sample had not been acidified. Add 2 drops of 0.2M BaCl<sub>2</sub>. If a precipitate is produced, isolate the precipitate and treat it with 6M HCl. Look for bubbles and note if any precipitate remains.

### ***Procedure 24***

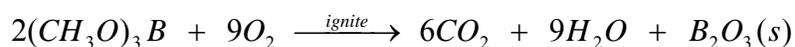
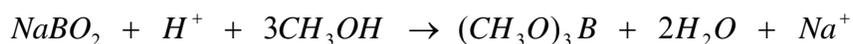
Treat a small pinch of the solid unknown to be tested with 3 drops of 18M H<sub>2</sub>SO<sub>4</sub>. Look for bubbles, color in the solution, color in the gas, and test the gas with wet blue litmus. If strongly acid, colored gases are not observed, carefully heat the mixture over a small flame. Now yellow-brown gas on heating indicates nitrate. These latter tests are usually feasible only if no strongly acidic gas producing anions are present.

## **Specific Anion Procedures**

### ***Procedure 25 Test for Borate Ion***

With a small quantity of the solid salt to be tested for borate in a casserole, add 3 drops 18M H<sub>2</sub>SO<sub>4</sub> (hood) and 20 drops of methanol. Heat this mixture over a burner in the hood about 5 seconds then set it afire by tipping the casserole so the flame lights the fumes. The immediate appearance of a green flame indicates the presence of borate ion. Though the cations Ba<sup>2+</sup> and Cu<sup>2+</sup> have distinct green flames they are not volatile and don't give the immediate color.

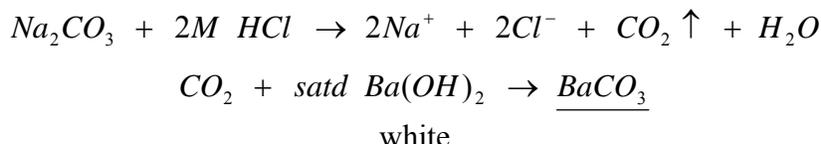
Equations for Procedure 25



### ***Procedure 26 Test for Carbonate Ion***

Place a small amount of solid sample of the salt or mixture to be tested in a dry test tube. Get about one mL of saturated Ba(OH)<sub>2</sub> from the reagent shelf. If this solution is not clear, centrifuge it and draw some up in your Pasteur pipet. Now, squirt a portion of 2M HCl onto the solid you are testing. If carbonate is present there will usually be vigorous effervescence. Insert the Pasteur pipet about half way into the tube and squeeze out a drop so that it hangs on the pipet. Immediate clouding of the suspended drop implies the presence of CO<sub>2</sub>, thus carbonate. If any Ba(OH)<sub>2</sub> is dropped in the solution it doesn't matter.

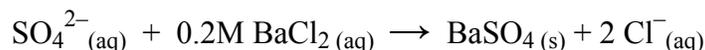
Equations for Procedure 26



### ***Procedure 27 Test for Sulfate Ion***

Use a few drops of the solution to be tested and acidify it with 6M HCl. Add one or two drops of 0.2M BaCl<sub>2</sub>. A white precipitate indicates the presence of sulfate ion.

Equation for Procedure 27



### ***Procedure 28 Test for Chloride Ion***

Test for chloride by first acidifying a 5 drop sample with 3M HNO<sub>3</sub>. Add two drops of 0.2M AgNO<sub>3</sub>. A white curdy precipitate proves the presence of Cl<sup>-</sup>.

Equations for Procedure 28



### ***Procedure 29 Test for Nitrate Ions***

Use only 2 drops in the nitrate test and add 10 drops of 18M H<sub>2</sub>SO<sub>4</sub> and mix. Now carefully layer a few drops of freshly prepared FeSO<sub>4</sub> solution—do not mix. After a few minutes, you will observe a brown discoloration at the interface between the heavy 18M H<sub>2</sub>SO<sub>4</sub> and the lighter FeSO<sub>4</sub> solutions. The brown species is Fe(NO)<sup>2+</sup>, nitrosyliron(II) ion, in solution.

Fresh FeSO<sub>4</sub> solution — put a pinch of FeSO<sub>4</sub>·7H<sub>2</sub>O in a test tube and add a few drops of 3M H<sub>2</sub>SO<sub>4</sub> then add about 2 mL of water. Stir until dissolved.

Equation for Procedure 29

