**Qualitative Analysis for Fe3+, Ba2+, Ni2+, Pb2+, and Ag+**

How do you know what is in a sample? Modern instrumental methods allow the composition of a sample to be determined quite accurately by simply injecting some of the sample into an instrument, but many of these instruments are fairly slow and large which prevents their use outside a laboratory setting. How can a geologist determine the type of rock she finds in the field? How can a police officer decide whether the white powder found in a suspect’s car is baby powder or illegal drugs?

Before the advent of modern instrumentation, chemists were forced to rely upon their observations of physical properties and reactivity, and had to use those observations in a logical progression to determine the composition of an unknown sample. In this experiment, we will be exploring the behavior of a variety of metal cations (positively charged ions). Using our observations, we will identify an unknown sample

You have been provided with solutions of the nitrate salts of the 5 cations listed in the title of the experiment. You will test each of these cations under a variety of conditions as outlined below to observe their behavior under a variety of conditions. Your observations should include:

1. Formation of a precipitate

2. Color of any precipitate that may form 3. Evolution of a gas

4. Dissolution of a precipitate

5. Changes in the color of the solution

Your ability to correctly identify your unknown is dependent upon your observations, so record everything you see! Your observations will also depend very strongly upon the quality of the reagents you use to perform these tests. The supplied reagents are clean, but it is very easy to cross- contaminate them with poor lab procedure. When dispensing reagents into a test tube with a dropper, the tip of the dropper must NEVER touch the test tube. Always release the drop from a little above the mouth of the test tube.

Once the two solutions have each been added to the test tube it is important to thoroughly mix the solutions in order to get the correct result. Hold the top of the test tube firmly between two fingers. With your other hand pull strongly on the bottom of the test tube and then slide this hand off the bottom of the tube. If you’re holding the top of the tube firmly this should create a strong mixing action in the tube without splashing any solution out of the tube.

Lastly, be careful to minimize waste in the experiment. Empty your tubes in a waste beaker at your lab bench. Use only a few drops or, at the most, a few mL of water to rinse the contents of the tube into your waste beaker. If it does not come clean with a few small rinses don’t keep rinsing. You’re only adding water to the waste and increasing the cost of waste disposal. Once the tube is emptied it can be washed with a brush and soapy water in the laboratory sink.

Safety Concerns: Most of the chemicals we are using today are not hazardous; however, you must always exercise caution when performing any experiment.

You must ALWAYS wear your safety goggles while the lab. Even if you aren’t performing an experiment, your neighbor might be and there is always the possibility that a chemical can splash toward your face. ALWAYS wear your goggles!

Strong acids and bases can burn your skin, but all of the acids and bases we will be using can be safely washed off with water. If you get any reagent on your skin, wash the area thoroughly with water at the sink.

Both ammonia and hydrochloric acid have VERY strong odors. When using these reagents, avoid smelling them as much as possible

1

Experimental Procedure:

You do not have to do these tests in numerical order, but once you start doing a number, do all parts of that number in order. For example, you can do #5 first, but you must do 5a then 5b then 5c.

1. To 10 drops of fresh test solutions (the nitrate solutions of the five cations listed in the title, each in its own test tube), add 10 drops of 15M NH3(aq) dropwise.

2. To 5 drops of fresh test solutions, add 2-3 drops of K2CrO4(aq).

3a. To 10 drops of fresh test solutions, and add 5 drops of 3M H2SO4 to each. Record any observations.

3b. If a precipitate forms, centrifuge the mixture. Decant the supernatant; that is, carefully pour the supernatant out of the tube leaving the precipitate behind. Wash the precipitate once by adding about 2mL of distilled water, mixing, centrifuging and discarding the wash liquid. To the precipitate, add 6M HNO3 (10-20 drops) to see if the precipitate dissolves.

4a. To 10 drops of fresh test solutions, add 5 drops of 6M HCl. If a precipitate forms, mix the sample thoroughly, centrifuge, and discard the supernatant. Wash the precipitate with about 2mL of water, centrifuge and discard the supernatant.

4b. To any precipitates formed in 4a, add about 3mL of fresh distilled water. Heat the tube as directed by your lab instructor. If a precipitate remains when the sample is hot, quickly centrifuge and decant the hot liquid into a separate tube. (Save the precipitate for Part 4d.)

4c. To the hot liquid that was decanted in Part 4b or to any solutions from Part 4b where the solid completely dissolved, add 1-2 drops of K2CrO4(aq) and mix thoroughly.

4d. Wash any precipitate remaining from Part 4b with about 2mL of distilled water. Centrifuge and discard the wash liquid. Add 5-10 drops of 15M NH3(aq) to the precipitate, mix well and observe any changes in the precipitate and the supernatant.

5a. To 10 drops of fresh test solutions, add 10 drops of 3M NaOH one drop at a time, mixing and observing the sample carefully after every drop. Centrifuge any samples that have precipitates after all 10 drops are added, discard the supernatant and save the precipitate for part 5b.

5b. Wash the precipitates from Part 5a once with water, centrifuge and discard the supernatant. To the precipitate, add 10 drops of 15M NH3(aq) and mix thoroughly.

6a. To 10 drops of fresh test solutions, add 10 drops of 3M NaOH. Centrifuge and decant the supernatant, saving the precipitates for Part 6b.

6b. Add 5-10 drops of 6M HNO3 to the precipitate and mix thoroughly.

6c. Add 2 drops of 1M NH4SCN solution to each of the solutions from Part 6b.

Week 1: Practice experimental procedures for analysis of “known” cations.

This first week you will be carrying out the experimental procedures described above on solutions of “known” cations. This will give you practice observing the results and give you information for writing the net ionic equations and constructing a flow chart. We will review your results and the flow chart that will be most useful in separating all of the cations from one another as a class. The handin for this first week will be writing all of the net ionic equations for the observed positive reactions. This will be a ten point assignment.

Chemical Equations:

Give net-ionic equations to represent only those species that are reacting in each case. Be sure that your equations are balanced. The following guidelines will enable you to make accurate predictions of the products in most cases.

2

1. Assume that most reactions result from a simple combination of cations and anions.

2. If the product is a solid assume that it is electrically neutral, that is, the total cationic (positive) and anionic (negative) charges in the solid compound are equal.

3. If you have made an observation that indicates that a reaction has occurred and the product is in solution assume that it is not necessarily electrically neutral but may possess a net charge, which may be either positive or negative.

Week 2: Analysis of an Unknown:

Each person will be assigned an unknown to analyze. The unknown you are given will contain a mixture of the metal cations you have tested. Given the results and observations you have collected with the known samples, devise a plan (a flow chart), which will allow you to separate and identify each of the metal cations that are present in your mixture. Your unknown could contain as few as 1 cation or as many as 5, so your flow chart should allow you to independently and positively verify the identity of each of the 5 cations and physically separate these 5 cations from each other. This means that in many cases negative results will not be useful, so be very careful! Your flow chart showing the results of your unknown analysis should be recorded in your lab notebook.

3