Qualitative Analysis of Anions

Introduction:

Chemists use a wide variety of methods to characterize the chemical species in a substance. Generally, such methods are divided into two categories: ***quantitative analysis*** focuses on determining the amount (the quantity) of such chemical species, while ***qualitative analysis*** focuses on identifying the chemical species based on their chemical and physical properties.

Your goal in this lab is to perform a series of *qualitative* chemical tests – based on chemical reactivity and solubility – to determine the identity of anions in an aqueous solution of unknown composition. Your unknown solution will contain **2, 3, or 4** of the following anions: *chloride*, *carbonate*, *iodide* and *sulfate*. As these ions are the ones being analyzed, they are referred to as the *analytes*.

This laboratory experiment is a little different from the others that you have conducted. It is intended to provide minimal step-by-step directions, challenging you instead to engage in guided inquiry as you apply your growing chemical skills to the problem before you. In the first week, you will work *with your team* to develop procedures and test them on a solution known to contain all four anions. In week two, you will work *individually*, using the procedures your team developed in order to identify the anions present in two different solutions of unknown composition.

Materials:

Single-reagent analyte solutions:

- 0.20 M NaI (aq)
- 0.20 M NaCl (aq)
- 0.20 M Na_2SO_4 (aq)
- 2.0 M Na_2CO_3 (aq)

Multiple-reagent analyte solutions:

- **Known**: aqueous solution containing a mixture of equal volumes of all four analytes
- **Unknown A**: aqueous solution of only **two, three or four** of the four analytes
- **Unknown B (numbered)**: aqueous solution of only **two, three or four** of the four analytes

Testing reagent solutions:

- 6 M HNO_3 (aq)
- 0.2 M $Ba(NO_3)_2$ (aq)
- 0.2 M $AgNO_3$ (aq)
- 6 M NH_3 (aq)
- 15 M NH_3 (aq)

Other materials:

- Litmus paper (red and blue)

Procedure:

WEEK ONE:

Experiment PART A: Your group's goal is to develop a set of experimental procedures to identify the presence of all four anions in the KNOWN aqueous solution containing all four anions. It has been made by combining equal volumes of 0.20 M aqueous solutions of NaCl, NaI and Na_2SO_4 and 2.0 M Na_2CO_3.

To simplify your tests, start by testing each ion separately to make sure you know what it looks like when you get a positive result. React each individual ion solution with the appropriate reagent and record your observations.

Remember: in order to accurately compare precipitates, you will need to use the same concentration of solutions. Since the combined KNOWN solution contains 4 different solutions, you should use water to dilute each separate ion solution so that the ion has the same concentration it would have in the KNOWN mixture. The KNOWN solution has 4 parts by volume. Parts can be measured in drops or milliliters. Therefore, to test a single ion, a solution should be made with 1 part of the solution and 3 parts of water. If you use 5 drops as one part, then you would use 5 drops of the ion and 15 drops (5 drops x 3 parts) water.

After you are familiar with the positive results for each separate ion test, you should test the KNOWN mixture of four ions. Use the flow chart provided to assist you in your experimental design to separate all 4 ions in the KNOWN solution. Please consider that every step in your flow chart is considered destructive to your sample, and that you have a limited amount of sample available. Consequently, your tests should be conducted with the smallest amount of sample possible that will produce results that you will be able to see. Start with 1 mL (20-25 drops) of the KNOWN solution.

Once you have developed a draft of your procedural design, your team should test it on the KNOWN solution to confirm that it works. Make sure that you take notes as you go ***in the Observation Tables*** *(in your handout)* so that you are confident you can follow this procedure on your own with an unknown next week! It might also be very helpful to duplicate the flow chart and make notes on it for future reference.

Some helpful notes on important theory and techniques used in this experiment:
This link is a helpful resource for techniques:
http://www.public.asu.edu/~jpbirk/qual/qualanal/semimicr.html

This link is a helpful resource for reactions and equations of anions:
http://www.public.asu.edu/~jpbirk/qual/qualanal/anprop.htm

1. **Solubility**. The positive test for most of the anions in this experiment is the formation of a sparingly soluble precipitate. As you have learned when studying the solubility product, $\boldsymbol{K_{sp}}$, even salts that are often considered "insoluble" are soluble to some degree. Indeed, if sufficient water is added, any precipitate would dissolve and become invisible to the eye, thereby imitating a negative test result. (Consider how $Q < K$ because the concentrations of these solutions are so low.) Take care to not add more reagent than absolutely necessary when you run your tests. That way, your precipitates will be readily visible. Remember: a precipitate may appear as a solid deposited at the bottom of a test tube, or it may appear as cloudiness, when the precipitate is suspended in solution and hasn't settled yet.

2. **Concentrations**: Because concentrations change when other solutions (not just pure water) are added, we have to calculate concentration when making solutions of individual ions. If we start with 2 mL of 0.20 M NaCl, its concentration changes after we add 2 mL of the other 3 solutions. Therefore, when testing an individual anion solution, the concentration must be made to the same level as the known solution so observations of reactions match.

3. **Testing for pH:** The general technique to test whether a solution is acidic or basic is to use litmus paper. One piece of litmus paper can be torn into 4 or 5 small pieces and placed on a paper towel. A small stirring rod is dipped into the solution to be tested and placed on the appropriate color litmus paper. Blue paper will turn red for acidic solutions; red paper will turn blue for basic solutions. Afterward, the stirring rod should be rinsed into the waste beaker with D.I. water.

4. **Sample preservation.** You will have about 10 mL of the known solution (containing all 4 anions) to use in the first week. However, each step in the procedure is considered destructive to the sample. You will want to start with a small sample (about 1 mL) first so you have enough to run through the procedure several times and correct any mistakes that are made. You can calibrate a 1 mL sample by counting the number of drops it takes to reach the 1 mL mark on a graduated cylinder.

5. **Dropwise addition.** When adding solutions dropwise, you want to add as little solution as possible to get complete reaction. Remember that as you add solution, you are changing the concentration of the ion. When starting with 1 mL of solution, it is best to start by adding 3-5 drops of reagent, shake/mix the contents thoroughly. Then add 1 drop at a time; if no more reaction is observed with the addition of one drop, no more reagent needs to be added. Always keep track of the number of drops you add and record it so you can replicate your process.

6. **Centrifuging.** Keep in mind that the centrifuge must be balanced when in use. A test tube with an equal quantity of water should be placed opposite any sample being centrifuged to balance it. The lid should be closed while the centrifuge is spinning. Do not leave the centrifuge unattended when in operation, as even balanced centrifuges can wobble slightly and "creep" off of the benchtop. Because the number of centrifuges is limited, please share the centrifuge with other teams, keeping track of whose test tubes in the centrifuge are whose, by labeling them well.

7. **Complex ions.** Many polyatomic ions are formed with a metal atom in the center, surrounded by one or more groups (called *ligands*) of molecules or other ions that can be considered to be covalently bonded to the metal. Examples of such complex ions include the $Al(H_2O)_6^{3+}$ cation and the $CuCl_4^{2-}$ anion. The formation of complex ions often explains the unexpected solubility of certain metal-containing compounds. One complex ion you will encounter in this lab experiment is $Ag(NH_3)_2^+$, a soluble cation with two ammonia molecules as ligands. It is formed when certain silver salts are added to some solutions containing ammonia. Keep an eye out for when such a complex ion might be forming in your experiment!

WEEK TWO:
Experiment PART B: Using the experimental design your team developed and tested in week one, work ***individually*** to perform a qualitative analysis on **Unknown solution A** (to be provided) to determine which anions are present in that solution. When you are confident that you have identified the anions present, check your results with the instructor, who will give you the correct answer. Based upon your results (which will not be graded) re-test, adapt, and refine your procedure as needed.

Experiment PART C: Using the experimental design you have developed and refined, work ***individually*** to perform a qualitative analysis on a **numbered Unknown solution B** (containing **2, 3, or 4** of the four anions), to determine which anions are present in that solution. Each student will use a different unknown number. Your results from this test will be a significant part of your grade for this lab experiment, so perform any additional tests you feel appropriate to confirm your results before you are done. Be sure to record your unknown number at the top of page 9.

Clean-Up: Nitric acid, $HNO_{3\ (aq)}$, is a strong acid and is a corrosive and toxic substance that can cause severe burns and discoloration of the skin. **Concentrated aqueous ammonia, $NH_{3\ (aq)}$,** is a caustic base and a skin, eye and lung irritant. **Silver nitrate, $AgNO_{3\ (aq)}$,** can also stain the skin or clothing. Follow scrupulous safety protocols and avoid contact with your skin, eyes, and clothing and avoid breathing the fumes. In case of spills, wash affected area **immediately** with cold water and notify your instructor.

*****A note on interpreting the Experimental Flowchart for the separation of analytes*****

On the following page, you will find a flowchart depicting the various steps that can be taken to separate each analyte from solution. In the flowchart, the boxes represent the analytes and the products that result from reacting the analytes. The substances outside of the boxes represent the testing reagents that must be added ad a particular step to conduct a particular reaction.

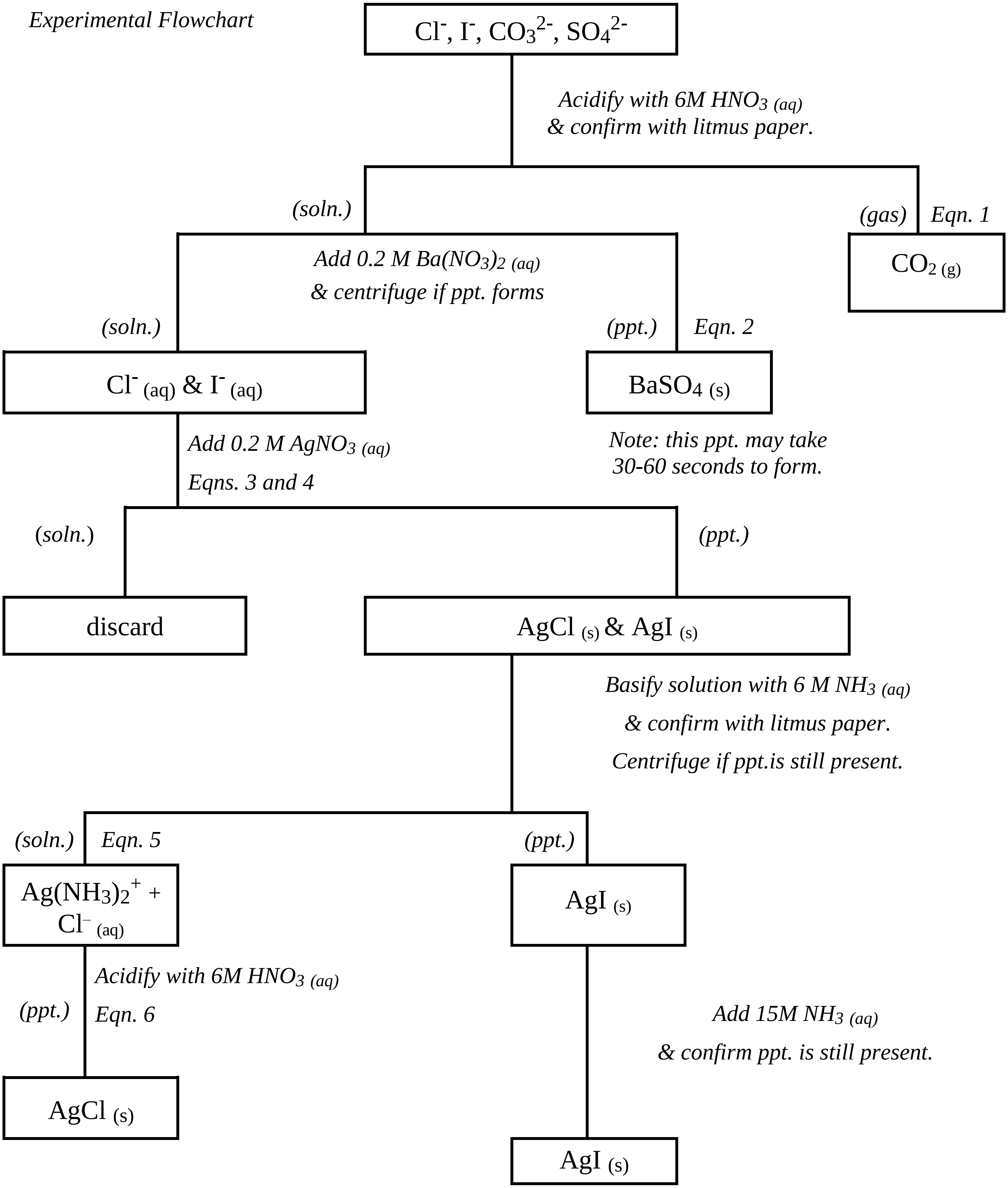
For example, in the flowchart you will see that all four analytes are featured in the top box. This represents the **Known** solution containing all four analyte compounds. Just beneath the box is text that indicates that you will add 6 M HNO_3 (aq) until it is acidic. So, this means that you would add the acid dropwise to a sample of Known solution and test for acidity after each drop. Once it is acidic, you stop adding acid.

Notice that, from there, the flowchart splits into two columns. This means that there is a separation possible at this point. On the left is the remaining solution, while on the right is the evolution of CO_2 gas from the solution. This gas will only form IF the appropriate analyte ion is in the solution. Therefore, seeing the gas is a positive confirmation of the presence of that analyte ion. Which analyte ion causes the generation of carbon dioxide when it is acidified? A hint is provided ("Eqn. 1") that refers to your Pre-Lab equations. When that analyte is reacted away, what is remaining in the Known solution?

Some flowchart steps show the chart splitting into a precipitate (ppt) and the remaining solution. When you see this, it means that the results must be centrifuged, and the supernatant solution decanted to leave the precipitate behind. Great care must be taken at these steps to not leave supernatant solution in the test tube with the precipitate, nor to allow the precipitate to transfer over with the supernatant decanted. Failure at this point could contaminate future tests and give false results.

In your **Unknown** solutions (A and B), of course, you will only have **two, three or four** of the four known analytes. So, one or more of these tests would be expected to give negative results. The skill with which you design your procedure and the care with which you carry out each step and make observations will determine whether you correctly identify which analytes are present and which are absent in your Unknowns.

Experimental Flowchart



Name: ___________________________________

Qualitative Analysis of Anions Pre-Lab Questions

Balance the following reactions and select the correct products for the Pre-Lab Quiz in Canvas. Note: The question numbers in canvas differ from those below because one of the reactions was not included in the canvas quiz.

Predict products for the following reactions. Be sure to include physical states and balance the equations.

1. ___ Na_2CO_3 (aq) + ___ HNO_3 (aq) → _____________________________________

2. ___ Na_2SO_4 (aq) + ___ $Ba(NO_3)_2$ (aq) → _____________________________________

3. ___ NaCl (aq) + ___ $AgNO_3$ (aq) → _____________________________________

4. ___ NaI (aq) + ___ $AgNO_3$ (aq) → _____________________________________

You will need to reference your flow chart to correctly predict products for # 5 and #6.

5. ___ AgCl (s) + ___ NH_3 (aq) → _____________________________________

6. ___ $Ag(NH_3)_2^+$(aq) + __ Cl^-(aq) + __ HNO_3(aq) → ___________________________________

Name: ______________________________ Partners: _________________________

Qualitative Analysis of Anions Lab Report

Data and Observations

I. Detailed Procedural Steps with Known Solution:

Table 1: Observations of each reaction of the Known Solution

II. Detailed Procedural Steps with Unknown A Solution:

Table 2: Observations of each reaction of Unknown A Solution

III. Detailed Procedural Steps with Unknown B Solution (number: ________)

Table 3: Observations of each reaction of Unknown B Solution

Results

Anions in **Unknown Solution A**:
Anions in *numbered* **Unknown Solution B**:

The Formal Report: Each student will write an ***individual, typed, formal report, worth 50 points***. Your report should be double-spaced, with 12-point font.

The report should include the following sections, clearly labeled:

A. (4 pts) A detailed **Purpose** stating the ions used in the *Known* and the purpose of both weeks 1 and 2.

B. (12 pts) A numbered, detailed **Procedure** based on your *Known* solution from week 1. Sub-bullets should be included to identify any differences made to procedure in week 2 with your *Unknown B*.

C. (6 pts) **Observations** of the individual ion reactions, the combined *Known* solution, and observations of your *Unknown B*. Your observations of each can refer back to your numbered procedural steps for clarity. (You do not need to repeat your procedure in this section.)

D. (4 pts) Write the **6** balanced equations for the reactions indicated in the flowchart.

E. (24 pts) A **Conclusion**, stating the *Unknown B* ***number,*** and the analyte anions present and absent. Discuss at least 2 sources of error. Explain how each error would affect your results, and describe how you would correct them if you were to repeat the experiment.

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Post-Lab Discussion Questions – Do not include these in your lab report! These questions will not be graded as part of your lab report grade. You will be responsible for the information in these questions and able to answer these or similar questions on the *post-lab quiz at the start of next week's lab period.*

1. Calculate the diluted concentration of ***each*** anion in the mixture after equal amounts of the anion solutions are all combined to make 1 mL of the *Known* solution. (Refer to page one of this handout for their initial concentrations.)

2. A student performing this lab experiment receives her *Unknown* solution and begins to conduct tests to identify the anions present. The acidification with nitric acid generates bubbles. The addition of aqueous barium nitrate to the remaining solution yields no precipitate. The addition of aqueous silver nitrate forms a precipitate. This precipitate is then basified with 6M aqueous ammonia and the precipitate completely dissolves. The resulting solution is re-acidified with nitric acid and a white precipitate forms. Which ***anions*** were present in the student's unknown? Explain.

3. A different student is running this same lab experiment. He first adds a few drops of nitric acid to his solution and generates some bubbles. He then adds aqueous barium nitrate solution and generates a white precipitate. From these results, he concludes that his unknown contains both carbonate and sulfate ions. However, his unknown actually contains no sulfate! Explain his error.

4. You are performing a different experiment with a solution that contains Na_2SO_4 and NaOH. Describe how you would separate the two anions (sulfate and hydroxide) and confirm the presence of each. Answer the following to help you design your procedure:

   a. What reagents could you add to precipitate each one?
   b. Which reagent should be added first and why?
   c. Write the equations for the reactions that would take place, and use this link (http://www.public.asu.edu/~jpbirk/qual/qualanal/catprop.html) to describe the observation you would expect to see to confirm the presence of each ion.
   d. Clearly describe the procedure you would follow to separate the two anions.