QUALITATIVE ANALYSIS

You will be working on your own (no partners) this week. The objective of this laboratory is to determine the identity of both the cation and the anion that are present in an ionic compound. This lab will be graded: 4 points each correct cation, 4 points each correct anion, 1 point each correct chemical formula for the unknown ionic compound, and 2 points for **observations**.

Introduction

Each of your two unknowns will consist of one cation and one anion. The possible cations are NH_4^+, Ca^{2+}, Mg^{2+}, K^+ and Na^+. The possible anions are Br^-, CO_3^{2-}, Cl^-, I^- and NO_3^-.

During the course of this lab, you will be asked to perform the following tests/procedures. Read them over now so that when you are asked to do them you know what needs to be done.

Flame Test

Acquire a nichrome wire with a small loop at one end. Heat the loop in the hot portion of a Bunsen burner flame located at the top of the light blue portion of the flame. The wire should glow red and a slight orange color should be visible in the flame. If this is not the case, dip the nichrome loop in 6 M HCl and reheat in the flame. Repeat this process until no color other than that from the nichrome wire appears in the flame. Once this is accomplished, wet the loop of your nichrome wire with deionized water and position the solid you wish to test in the loop. Place the loop into flame and note the color of the flame. Clean before next use.

Litmus Test

To test the pH of a solution, dip a piece of litmus paper into the solution. Red litmus paper changing to a blue color indicates the solution is basic. Blue litmus paper changing to a red color indicates the solution is acidic.

Centrifuge

A centrifuge is used to separate a solid from a liquid solution. To properly use a centrifuge, care must be taken to balance the rotor of the centrifuge. This is achieved by placing a test tube with a comparable volume of liquid in compartment opposite of your sample. Failure to do this will result in unnecessary wear on the equipment and cause the unit to vibrate. The sample should spin in the centrifuge for approximately two minutes.

Procedure

Identification of the Cation

Weigh approximately 0.25 grams of your unknown into a small test tube. Dissolve the sample in 2 mL of deionized water. To this solution add 2 mL of 6 M NaOH. Look for the formation of a precipitate. The formation of a thick white precipitate indicates the presence of calcium. Whereas, the formation of a faint white gelatinous precipitate indicates the presence of magnesium. If a precipitate forms, centrifuge your sample and decant off the supernatant. Note the description of the precipitate.

On those samples which precipitate, perform a flame test on your original unknown solid sample as explained in the introduction. A red color confirms the presence of calcium in the sample. Lack of a red and the presence of a white or bluish color indicates the presence of magnesium.

If no precipitate forms, dampen a piece of red litmus paper with deionized water. Heat the test tube containing the solution in a hot water bath while holding the dampened litmus paper over the mouth of the test tube. Be careful not to touch the test tube with the litmus paper. If the litmus paper turns blue, this indicates the presence of ammonium.

If all the preceding tests have been negative, perform a flame test on your original unknown solid sample. A purple flame confirms the presence of potassium. A brilliant orange flame confirms the presence of sodium.

Identification of the Anion

Weigh approximately 0.25 grams of your unknown into a small test tube. Dissolve the sample in 2 mL of deionized water. Check to see if the solution is basic. If it is not basic, add 6 M NH_3 one drop at a time until the solution is basic to litmus. Next, add 5 drops of 1 M $Ba(NO_3)_2$. Look for the formation of a precipitate.

If a precipitate forms, in all likelihood carbonate is your anion. To confirm this, add 5 drops of 6 M HNO_3. Obvious bubbling confirms the presence of carbonate.

If no precipitate forms, add drops of 6 M HNO_3 until the solution is acidic, testing with litmus paper. Add 5 drops of 0.2 M $Fe(NO_3)_3$ to the solution and ten drops of toluene. Swirl to mix. The toluene layer, the top layer, turning colored, indicates the presence of iodide. *To be sure that you are looking at only the toluene layer, pipette off the top layer and look at its color, then pipette the toluene back into the solution.*

If the toluene layer remains colorless, add three additional drops of 6 M HNO_3 and 5 drops of 0.1 M $KMnO_4$ to the solution. The toluene layer turning colored indicates the presence of bromide. *To be sure that you are looking at only the toluene layer, pipette off the top layer and look at its color, then pipette the toluene back into the solution.*

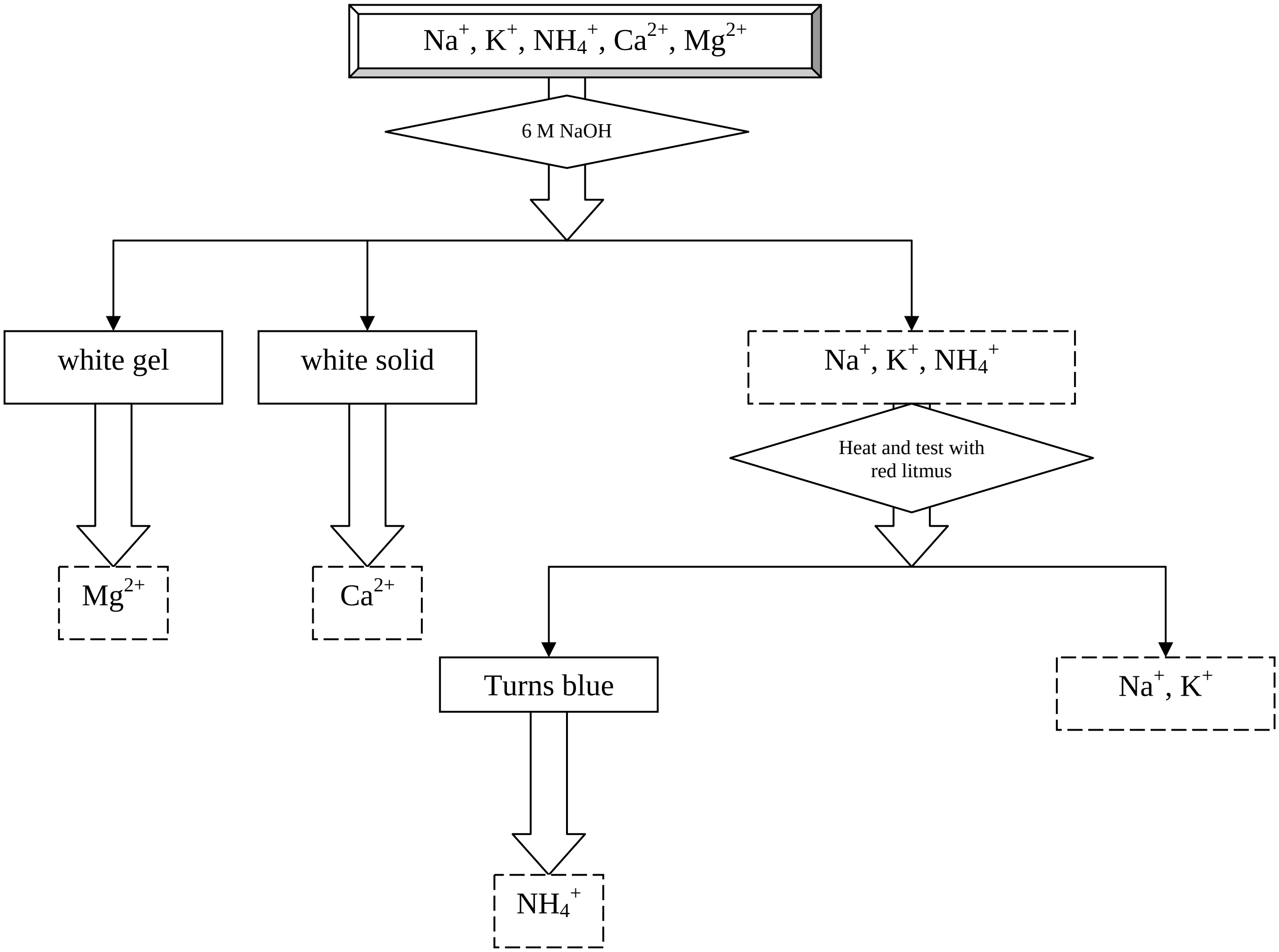
If the toluene layer still remains colorless, remove the toluene layer with a plastic pipette and dispose in the organic waste container. Next, add 15 drops of 0.1 M $AgNO_3$ to the remaining solution. Formation of a precipitate indicates the presence of chloride if you have done the previous steps correctly. To make sure, centrifuge your sample and decant off the solution. Your precipitate should be white if you indeed have chloride. A different colored precipitate would indicate that either the iodide or bromide tests were done incorrectly. Go back and redo these steps.

If no precipitate forms, your anion is most likely nitrate. Proceed with the following test to confirm nitrate presence.

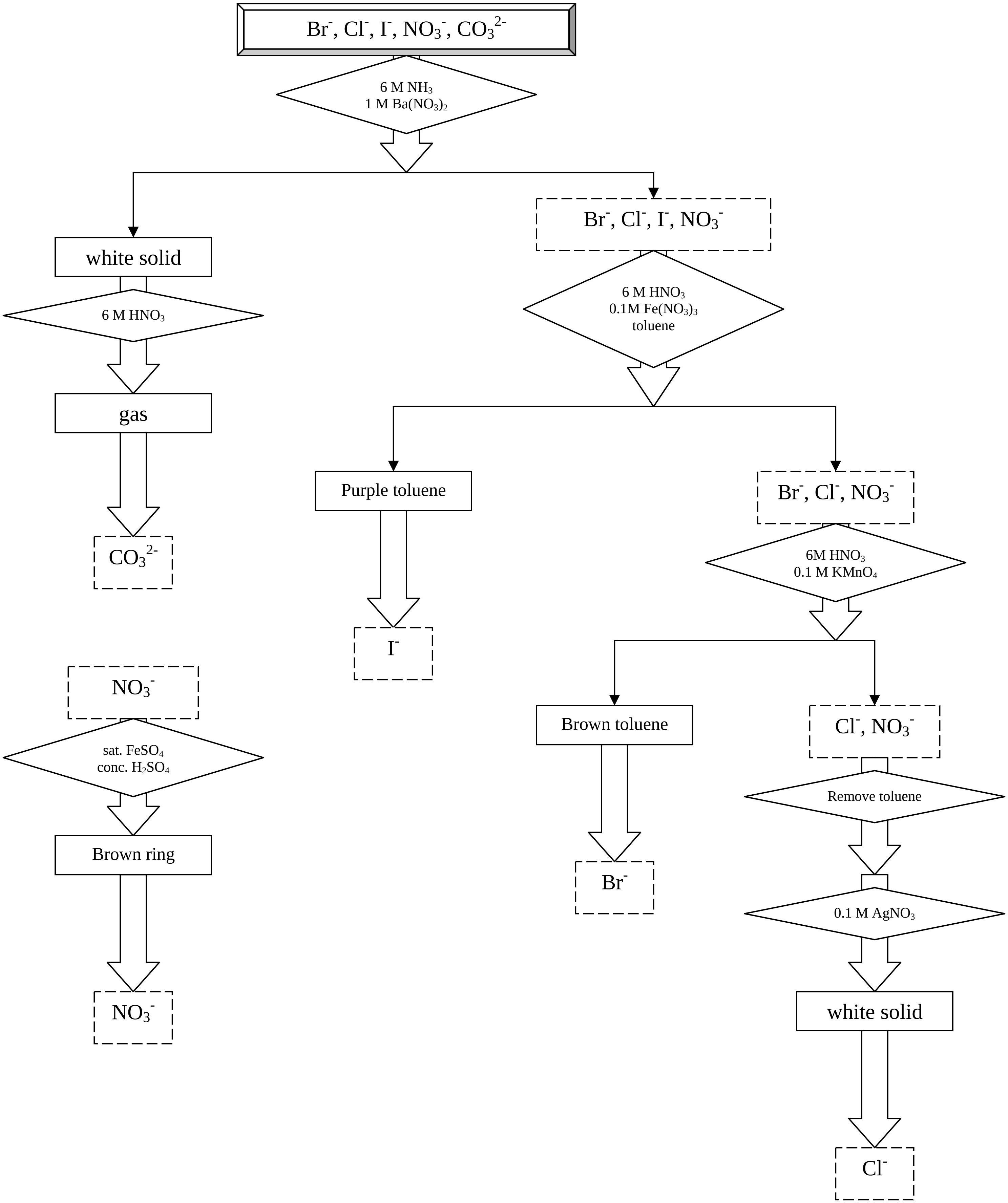
Weigh approximately 0.25 grams of your unknown sample into a small test tube. Dissolve in 2 mL of deionized water. In a separate test tube prepare a fresh solution of saturated $FeSO_4$ solution. This is done by weighing approximately 0.1 grams of $FeSO_4$ and adding 3 mL of deionized water. Stir for several minutes to insure that the solution is saturated. Allow the mixture to set to allow the solid $FeSO_4$ to settle. There should still be some solid visible in the bottom of the test tube. Next, add 0.5 mL of this saturated $FeSO_4$ solution to your unknown sample solution and stir. Place this solution in an ice bath for approximately two minutes. Remove your test tube from the ice bath and tilt to a 45° angle. Use a plastic pipette to slowly add 1 mL of concentrated H_2SO_4 drop-wise to the inside wall of your test tube. Extra care should be taken to insure that the concentrated H_2SO_4 and your solution do not mix. The H_2SO_4 should sink to the bottom of your test tube and you should be able to see two distinct layers. Allow your test tube to set for several minutes. A brown layer forming at the interface of these two layers confirms the presence of nitrate in your sample. (Note: If your cation is calcium, you will also see a white precipitate form. A brown layer at the interface should still be identifiable.)

Disposal

When you are finished with this lab, deposit the containers with the remainder of your unknowns in them in the box provided.



Flame Test on Unknown Solid	
Ca^{2+}	Red
K^{+}	Purple
Na^{+}	Orange


Br^-, Cl^-, I^-, NO_3^-, CO_3^{2-}
6 M NH_3
1 M $Ba(NO_3)_2$
white solid
6 M HNO_3
gas
CO_3^{2-}
Br^-, Cl^-, I^-, NO_3^-
6 M HNO_3
0.1M $Fe(NO_3)_3$
toluene
Purple toluene
I^-
Br^-, Cl^-, NO_3^-
6M HNO_3
0.1 M $KMnO_4$
Brown toluene
Br^-
Cl^-, NO_3^-
Remove toluene
0.1 M $AgNO_3$
white solid
Cl^-
NO_3^-
sat. $FeSO_4$
conc. H_2SO_4
Brown ring
NO_3^-

Name: ___________________________________

Data and Analysis Sheet: Qualitative Analysis
First Unknown

Unknown # ____________________

Description of unknown sample:

Identification of the Cation

Observations after adding 6 M NaOH:

If precipitate forms, what is its chemical formula? ____________________

Observations from flame test:

Observations from heating sample in the hot water bath:

If litmus turns blues, what gas is causing the color change? ____________________

Observations from flame test:

Identity of Cation: ____________________

Data and Analysis Sheet: Qualitative Analysis
First Unknown

Identification of the Anion

Observations after adding 1 M $Ba(NO_3)_2$:

If precipitate forms, what is its chemical formula? ______________________

Observations after adding 0.2 M $Fe(NO_3)_3$ and the toluene:

What is the color of the toluene layer? ______________________

Observations after adding 0.1 M $KMnO_4$:

What is the color of the toluene layer? ______________________

Observations after adding 0.1 M $AgNO_3$:

If precipitate forms, what is its chemical formula? ______________________

Observations from the nitrate test:

Identity of Anion: ______________________

Chemical Formula for First Unknown: ______________________

Data and Analysis Sheet: Qualitative Analysis
Second Unknown

Unknown # ______________________

Description of unknown sample:

Identification of the Cation

Observations after adding 6 M NaOH:

If precipitate forms, what is its chemical formula? ______________________

Observations from flame test:

Observations from heating sample in the hot water bath:

If litmus turns blues, what gas is causing the color change? ______________________

Observations from flame test:

Identity of Cation: ______________________

Data and Analysis Sheet: Qualitative Analysis
Second Unknown

Identification of the Anion

Observations after adding 1 M $Ba(NO_3)_2$:

If precipitate forms, what is its chemical formula? ______________________

Observations after adding 0.2 M $Fe(NO_3)_3$ and the toluene:

What is the color of the toluene layer? ______________________

Observations after adding 0.1 M $KMnO_4$:

What is the color of the toluene layer? ______________________

Observations after adding 0.1 M $AgNO_3$:

If precipitate forms, what is its chemical formula? ______________________

Observations from the nitrate test:

Identity of Anion: ______________________

Chemical Formula for Second Unknown: ______________________