Lab 11: Identification, Properties, and Synthesis of an Unknown Ionic Compound

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Discussion and Scientific Explanations

Quantitative Analysis

The unknown compound obtained was composed of white crystals and contained a slight "salty" odor according to our General Physical Properties description.

Solubility Tests

For the solubility tests, in water, the compound was completely soluble and had a pH of 4.5. This indicates that the unknown compound is slightly acidic and is a polar or ionic compound. According to the pH rating system, a pH of 0 is the most acidic while a pH level of 14 is the most basic. With a pH of 4.5 the unknown compound it can be concluded that the unknown compound is slightly acidic. The compound could be ionic because ionic compounds tend to be soluble in substances that have polar covalent bonds, like water². Knowing this, the unknown compound should not be soluble in toluene or acetone because that would test positive for nonpolar or not ionic compounds. When tested with toluene and acetone, they were not soluble as seen in **Table 1a**. When tested with 1 M NaOH and 1 M HCl both were soluble. This proved rather strange because since the pH tested to be acidic, the compound should dissolve in the 1 M NaOH. However, when mixed with 1 M HCl, the compound dissolved but there were still small traces of the compound at the bottom of test tube. One possibility of this error is because the ratio of 1 M HCl to unknown compound was large enough to dissolve the compound. To conclude the results, the unknown compound proved to be slightly acidic and possibly a polar or ionic compound.

Conductivity Tests

In **Table 2a**, the conductivity tests are shown with water and with water plus the unknown content. The resistance when tested with the compound went lower by 88.2 k Ω . This means that the unknown compound is very conductive to electricity. This further proves that the unknown compound is ionic because of ionic properties. While dry, ionic compounds tend to not conduct electricity well. However, when the ions are separated in solution like water, they conduct electricity well².

Anion/Cation Tests

The anion test helped to determine if certain ions were present in our compound. The ions that were tested for were chloride, sulfate, nitrate, carbonate, and acetate as seen in **Table 3a**. The unknown compound tested positive for both chloride and acetate. The chloride test formed a white precipitate when mixed. The positive result indicates that there is a presence of a chloride ion but also positive for sulfates or halogens¹. Since there were no tests for the iodide or bromide, which are halogens, they can be eliminated from our unknown compound. The next test was for the sulfate test. The test came out negative and didn't form any precipitate. This allows us to conclude from the chloride test that there are no sulfates, iodide, bromide or other halogens in our unknown compound. Therefore, it can be stated that chloride is our anion. The acetate produced a slight fruity smell and could have a possibility of have an acetate ion. However, the fruity smell could be from the ethanol itself because ethanol has a fruity smell to it. A strong fruity smell would indicate a

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presence of an acetate¹. The negative result for the nitrate test and carbonate test proved that indeed that our compound had a chloride anion. There was one error made for the nitrate test, which was re-done in order to correct it. The first time the test was done, there was no yellow color from the nitrate. It is possible there was a mix-up between using the concentrated $\rm H_2SO_4$ and 1 M $\rm H_2SO_4$. Our group was able to conclude that our compound contained a chloride ion.

The next step was to find the cation now that the anion was found. To do this, 1 mL of solution was added to 1 mL 6 NaOH and try to see if there is an ammonia smell. In this test, in **Table 4a**, it tested positive and had a strong smell of ammonia. Then next is for the flame test. Cations can be identified by the flame test because the excitation of an electron followed by a decay of the electron can emit a color of light. Several light colorations can be seen on page 63 in our lab manual¹. Our group concluded that there was no color emitted and tested negative for any of the elements listed except magnesium. Magnesium can be ruled out because of our positive result from the ammonium test. After investigating and performing these experiments, our group concluded that our unknown element is ammonium chloride (NH₄Cl). The tests were re-done with the ammonium chloride and had the exact same results as our unknown compound.

Reactivity Test

Now that the compound is classified, further tests can be done to prove that the compound is the correct. In **Table 5**, five sets of reactivity tests are done to determine the identity of the compound. By choosing the reactants and predicting the products, one can deduct if the reaction will create a reaction or not. To do this, double replacement reactions must be done. In double replacement reactions, the cations and anions of the two different compounds in the reactants switch places to form the products. The reactants(ionic compounds) exchange ions and form two new products with the same ions². The format is:

$$A^{+}B^{-} + C^{+}D^{-} \rightarrow A^{+}D^{-} + C^{+}B^{-}$$

Once the experiment has been done, a comparison of our predictions with the actual product can prove that the compound is the right one.

Reaction #1:

$$NH_4CI + AgNO_3 \rightarrow NH_4NO_3 + AgCI$$

Both ammonium chloride(NH₄Cl) and silver nitrate(AgNO₃) are both soluble. However, when combined the predicted the products will be a soluble(NH₄NO₃) and a insoluble(AgCl) compound. All chlorides(Cl) are soluble except Ag⁺, Hg₂²⁺, and Pb²⁺. All nitrates(NO₃⁻) are all soluble. The solubility of these compounds can be found in the lab manual on page 60^1 . This will form a precipitate because the AgCl is not soluble. Preforming this reaction, one can compare our predictions and actual reactions and further determine if our compound is correct. As expected when performed, according to **Table 5**, a milky white precipitate was formed.

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Reaction #2:

$$NH_4Cl + NaOH \rightarrow NH_3+H_2O + NaCl$$

In this reaction the ammonium chloride(NH₄Cl) and sodium hydroxide(NaOH) are the reactants. Then double replacement reaction is used and the product would be NH₄OH + NaCl. The formula was changed because of the ammonium and hydroxide. These two form an ammonia gas(NH₃⁺) and water. The sodium chloride is soluble because all chlorides(Cl) are soluble except Ag⁺, Hg₂²⁺, and Pb²⁺ and expected to have no precipitate in this reaction. In our actual reaction our group proved this reaction as seen in **Table 5**.

Reaction #3:

$$NH_4Cl + KOH \rightarrow NH_3^+H_2O + KCl$$

In this reaction the ammonium chloride(NH₄Cl) and the potassium hydroxide(KOH) are the reactants. Then double replacement reaction is used and the product would be NH₄OH + NaCl. However, this is like Reaction #2 in that the NH₄OH form an ammonia gas(NH₃+) and water. The potassium chloride is soluble because all chlorides(Cl) are soluble except Ag+, Hg₂²⁺, and Pb²⁺. So, there is no precipitant expected and was proven when the experiment was done.

Reaction #4:

$$NH_4Cl + MgOH \rightarrow NH_3^+H_2O + MgCl_2$$

In this reaction the ammonium chloride(NH₄Cl) and the magnesium hydroxide(MgOH) are the reactants. Then double replacement reaction is used and the product would be NH₄OH + MgCl₂. However, this reaction is like Reaction #2 and #3 and the NH₄OH is expressed as NH₃+H₂O because ammonia gas is expected with water. The MgCl₂ is soluble¹ because all chlorides(Cl) are soluble except Ag⁺, Hg₂²⁺, and Pb²⁺. This reaction would have no expected precipitants and the actual experiment confirmed it.

Reaction #5:

$$NH_4Cl + FeSO_4 \rightarrow (NH_4)_2SO_4 + FeCl_2$$

When this double replacement reaction is performed, the products contain ammonium sulfate (NH₄)₂SO₄ and iron(II) chloride FeCl₂. The ammonium sulfate is soluble because all sulfates except Sr^{2+} , Ba^{2+} , and Pb^{2+} are soluble¹. The iron(II) chloride is also soluble¹ because all chlorides(Cl) are soluble except Ag^+ , Hg_2^{2+} , and Pb^{2+} . Since both products are soluble, no precipitant should be expected. As seen on **Table 5** our results confirmed our statement.

The purpose of these reactions were to find out which reactions would or wouldn't form a precipitate when performed. This is important to identifying our unknown compound because if the predicted reactions are confirmed by the actual reactions, then

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one can conclude that the unknown compound is ammonium chloride. If the reactions didn't match that of our predicted reactions, then the identity of the compound is still unknown. The reactions proved that the claim that ammonium chloride is the unknown compound. The goal of identifying the compound is achieved.

Quantitative Analysis

Since the unknown compound is confirmed to be ammonium chloride in the qualitative analysis, the next step is to confirm the compound using quantitative analysis. To do this a reaction with a precipitant must be performed. Out of the 5 reactions, $NH_4CI + AgNO_3 \rightarrow NH_4NO_3 + AgCI$ was decided to be the best to perform.

With this reaction, a known mass of our unknown compound(NH₄Cl) can be used to determine the amount of precipitant(AgCl) will have in grams. To do this, **Figure 1** shows all the calculations. Using 0.2 grams of our unknown compound, the mol of NH₄Cl can be determined. To do that the 0.2 grams of NH₄Cl is divided by the molar mass(g/mol) found using the atomic weight of each element. From that the molar ratio of the NH₄Cl to AgCl is 1:1 and can conclude that the AgCl must have 0.00374 mol. According to the law of conservation of mass, during all transfers of matter and energy, the mass of the system cannot change over time and must be constant over time. The mass can be created nor destroyed but can be rearranged in space². Using 0.000374 mol of AgCl, it can be multiplied by the molar mass of AgCl to get the theoretical yield in grams. This number is 0.532 grams. So, before the experiment is done, it is known that 0.2 grams of NH₄Cl will produce 0.532 grams of AgCl.

Table 6 shows the results of three reactions performed. The ammonium chloride was mixed with hydrochloric acid in excess. The hydrochloric acid is the excess reactant, which means that the hydrochloric acid will remain because there is nothing with which it can react². The AgCl was removed from the test tube and onto a filter paper. That filter paper was weighed and subtracted from the total weight to get the actual weight of the AgCl. The average weight from the three was 0.533 grams. That is 0.001 grams difference from the predicted AgCl. The percent error of the experiment was 0.3%. This is a very positive result because not only does the qualitative analysis confirm our compound but also the quantitative analysis proves our compound is NH_4Cl .

Synthesis of Ammonium Chloride

Our goal of identifying they unknown compound was met. However, our next goal is to synthesized at least 5.0 grams of our compound in a way that will be cost effective, safe for the environment, and safe to perform.

Our group created three possible reactions to create ammonium chloride(NH₄CI):

- A. $NH_3 + HCl \rightarrow NH_4Cl$
- B. $(NH_4)_2CO_3 + HCl \rightarrow CO_2 + H_2O + NH_4Cl$
- C. BaCl₂ + $(NH_4)_2CO_3 \rightarrow BaCO_3 + NH_4Cl$

To figure out which reaction is the best to perform, the criteria of cost, safety, and environmental reactions are used to determine this. For reaction A, this is the simplest to Section 107

perform. However, the ammonia gas (NH_3) is hard to obtain and react with our lab and equipment. Reaction C has a number of problems. The barium chloride is slightly irritate to the skin, eyes, and lungs but the barium carbonate $(BaCO_3)$ is very toxic and is used in rat poison². This cannot be performed using our criteria because it will impact the environment and is not safe. The best reaction to perform is reaction B. The ammonium carbonate $[(NH_4)_2CO_3]$ and hydrochloric acid (HCl) is very common and cheap. The only downside of this is that the HCl is one of the strong acids². The product produced are the best for the environment. Carbon dioxide is found in our atmosphere and water harmless to humans and the environment.

To get 5.0g of our compound, the amount ammonium carbonate[(NH₄)₂CO₃] in grams must be found. The hydrochloric acid will act as the excess reactant just like in the quantitative analysis. **Figure 2** shows the calculations needed to get the amount of grams of ammonium carbonate. First the 5.0 grams of NH₄Cl is divided by the molar mass(g/mol) to get 0.0935 mol of NH₄Cl. This number is important to find the moles of (NH₄)₂CO₃ in the reaction. The ratio of moles of (NH₄)₂CO₃ to NH₄Cl is 1:2. This means that 0.0935 mol of NH₄Cl must be divided by 2 to satisfy the ratio. That gives 0.0468 mol of (NH₄)₂CO₃ in the reaction. To convert this to grams the 0.0468 mol of (NH₄)₂CO₃ is multiplied by the molar mass(g/mol). The result is 4.50 grams of (NH₄)₂CO₃ is needed to get 5.0 grams of NH₄Cl.

In the product of the reaction, carbon dioxide gas is released and water and ammonium chloride are left as the solution. As stated before, ammonium chloride is soluble in water. The problem is we needed to somehow get the ammonium chloride from the water. There is no precipitate that will form. To get the ammonium chloride, a recrystallization will be performed. Recrystallization is a technique that mixes the solution with hot solvent and cooled to form purified crystalline solid. The solvent used to perform the recrystallization will be ethyl acetate. The solid is precipitated and can be filtered off using vacuum filtration and oven heating treatment¹. If done correctly 5.0 grams of ammonium chloride will be obtained. To prove that it is ammonium chloride, the same qualitative and quantitative tests are performed with unknown compound as before.

Error Analysis

When performing experiments, errors can and will likely form. It is important to note these errors but also how they happened.

For this lab, there were not a significant amount of errors. One of the errors was in the anion tests. In week 2 of lab, the nitrate reaction was performed and produced a clear liquid and our group thought it had a negative reaction. However, when the anion tests were done with the known ammonium chloride sample, there was a yellowish brown color that formed but no brown ring at the junction of the liquids. This was still a negative reaction to the nitrate. But, it was performed with the unknown compound and had the same yellowish brown color as the ammonium chloride. A problem could have been when mixing the 3 mL of concentrated sulfuric acid, 1M of sulfuric acid might have been used. The lower molarity of the sulfuric acid(H_2SO_4) might have not been enough to react with our compound. Another error could have been that the mixture of the compound and the concentrated sulfuric acid(H_2SO_4) was not cooled properly before adding the 2 mL of

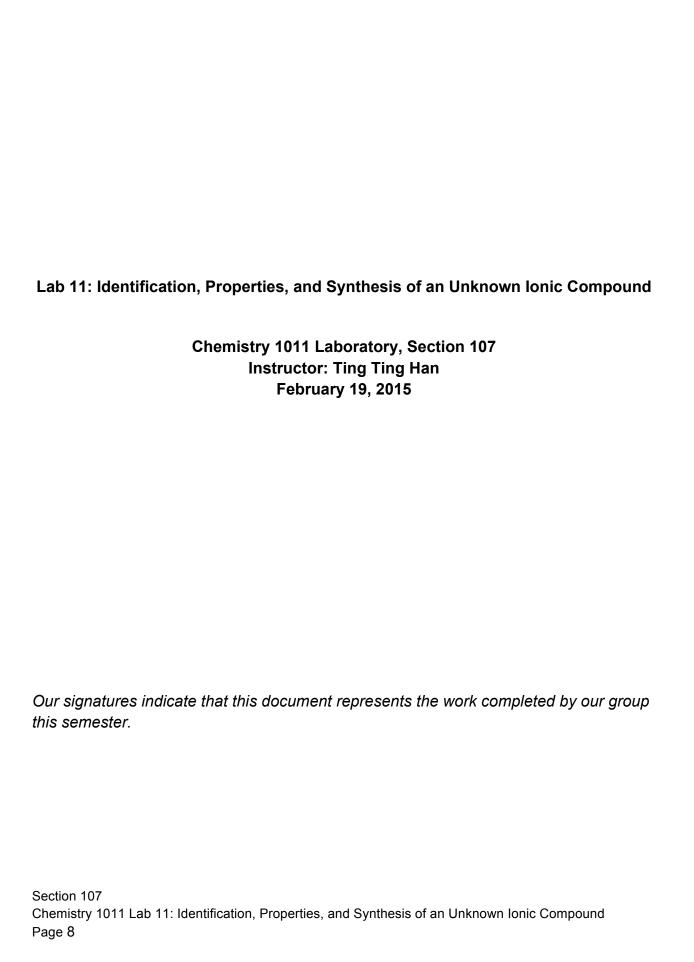
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ferrous sulfate(FeSO₄). This could alter the reaction with a higher temperature of the solution.

Another error was for the quantitative analysis. Although our percent error was 0.3% and the grams was close to 0.532 grams, when trying to get the AgCl out of the test tube, our group did not get all of the precipitate out and also ended up on our gloves or the table. This weight could or could not be substantial but the weight should have been a little more. That being said, our weight of the AgCl on **Table 6** was not only accurate but also precise. So, the precise weight could mean that our errors were negligible.

References

- 1. Cooper M. M., Cooperative Chemistry Laboratories, McGraw-Hill New York, NY, 2008.
- 2. Fay R.C., McMurry J.E., General Chemistry: Atoms First; Pearson: New York, 2014.



Goals

The goal of this laboratory project was to investigate an unidentified compound that had been discovered in a landfill in our hometown. Our group needs to identify the unknown compound. This includes finding as many chemical and physical properties of the compound to make predictions on how it will behave. Two syntheses of the compound will be prepared, compared for cost effectiveness, safety, and potential yield of the compound. Our group will be given no more than 5 g of the unknown compound to figure out the identity.

Experimental Procedures

Solubility Test

In order to determine what the unknown ionic compound is, there are numerous qualitative and quantitative tests ran in search for what exactly the unknown compound could be. The first test of the unknown compound is the qualitative solubility test. The solvents used were Water, Toluene, Acetone,. This experiment would reveal if the unknown compound is polar (or ionic) or nonpolar. Testing the pH of the soluble solution would also give more information about the unknown compound.

PH Test

To measure the pH of the unknown compound we mixed the compound with water. The pH was tested with a litmus strip and resulted in an orange color which indicates a pH level of 4.5.

Conductivity Test

Conductivity is a measure of how well a solution can handle electricity. A conductivity meter will be used to test this. Using a beaker, dissolve (if possible) the unknown compound in water and place the 2 probes in the water to determine the resistance in ohms. Make sure that the probes are in the water and not touching each other.

Anion Test

The next experiment performed on the unknown compound was the anion test. This test is performed to determine which of the anions are present in the unknown compound. The anions tested along with the unknown compound were Chloride, Sulfate, Nitrate, Carbonate and Acetate. All the individual anion tests performed needed to be tested with a soluble solution of the unknown compound. This amount of the unknown solution also depended on what anion it was being tested with.

The first anion which was Chloride, required 1 mL of the unknown solution with 1 mL of 6 M HNO3 and 1 mL of AgNO3. The second anion which was Sulfate, required 1 mL of the unknown solution with 1 mL of 6 M HCl and 1 mL of BaCl2. The third anion which was Nitrate, Section 107

required 1 mL of the unknown solution with 3 mL concentrated H2SO4 and then after this mixture cooled, pouring 2 mL of FeSO4 at a 45 degree angle into the mixture. The fourth anion which was Carbonate, required 1 mL of the unknown solution with 6 M HCl drop by drop. Finally, the fifth anion which was Acetate, required 2 mL of the unknown solution with 1 drop of concentrated sulfuric acid then adding 1 mL of ethanol in order for the solution to give off a fruity smell or not.

Cation Test

The cations in the unknown compound can be determined by precipitation of an insoluble salt. A test for ammonium was devised using 1 mL of 6 M NaOH. The resulting mixture was then smelled by wafting the air towards the nose. The smell of ammonia determines if there is a presences of ammonium ion. The test was also performed with the known NH₄Cl compound and had similar results from the unknown compound. Another cation test performed was the flame test. First, A nichrome wire was cleaned using heat by a Bunsen flame. The wire was dipped into the unknown compound and reheated. The flame was observed through a color filter to determine if there was flame coloration. If there was flame coloration, then the color would indicate the cation present in the unknown compound. A small sample of the unknown compound was placed on the nichrome wire, and then subjected to the flame. No color change was noted from the sample. The known NH₄CL returned the same results when subjected to the flame test.

Reactivity Test

After making deductions and gathering evidence, the unknown compound is now determined. To further investigate the conclusions, five reactions will be carried out to confirm the identity of the unknown compound. At least one of the five reactions must have a form of precipitate or chemical reaction. The reactions must be written out and determined if it will have an expected reaction or not. These reactions were chosen by first seeing what chemicals were accessible in the lab and because it was predicted that these reactions could form a precipitate with the unknown compound. The reactions were conducted by adding 5mL of a .1 molar solution of the unknown compound to 5mL of each of the following: AgNo₃, NaOH, KOH, MgOH, and FeSO₄. Refer to Table 5 for the reaction results.

Results

General Physical Properties: White, granular solid substance with slight "salty" odor

Table 1: Qualitative Solubility Test

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Table 1a. Unknown Compound Results:

Solvent	Solubility	
Water (pH = 4.5 with compound)	Soluble	
1 M NaOH	Soluble	
1 M HCI	Soluble (dissolved very quickly)	
Toluene	Not Soluble	
Acetone	Not Soluble	

Table 1b. NH₄Cl Results:

Solvent	Solubility	
Water (pH = 4.5 with compound)	Soluble	
1 M NaOH	Soluble	
1 M HCI	Soluble	
Toluene	Not Soluble	
Acetone	Not Soluble	

Table 2: Conductivity Test

Table 2a. Unknown Compound Results:

Solution KiloOhm (kΩ)	
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Water	90
Water + Unknown Compound	1.8

Table 2b. NH₄Cl Results:

Solution	KiloOhm (kΩ)
Water	88
Water + Unknown Compound	1.6

Table 3: Anion Test

Table 3a. Unknown Compound Results:

Anion	Change in Properties	Presence of ion
Chloride	Cloudy & White	Presence of Chloride ion or SO_4^{2-} , or CO_3^{2-}
Sulfate	No color change	No presence of ion, exclude ${\rm SO_4}^{2\text{-}}$
Nitrate	No color change	No presence of ion
Carbonate	No effervescing (bubbling or hissing sound) No presence of ion CO ₃ ²⁻	
Acetate	Very faint fruity smell Presence of ion	

Table 3b. NH₄Cl Results:

Anion	Change in Properties	Presence of ion
Chloride	Cloudy & White	Presence of Chloride ion or

		SO ₄ ²⁻ , or CO ₃ ²⁻
Sulfate	No color change	No presence of ion, exclude ${\rm SO_4}^{2\text{-}}$
Nitrate	No color change	No presence of ion
Carbonate	No effervescing (bubbling or hissing sound)	No presence of ion, exclude ${\rm CO_3}^{2-}$
Acetate	Very fruity smell	Presence of ion

Table 4: Cation Test

Table 4a. Unknown Compound Results:

Cation Test	Change in Properties	Presence of ion
Ammonium Test	Urine, ammonia smell	Presence of Ammonium ion
Flame Test	No color change	No presence of ion

Table 4b. NH₄Cl Results:

Cation Test	Change in Properties	Presence of ion
Ammonium Test	Urine, ammonia smell	Presence of Ammonium ion
Flame Test	No color change No presence of	

Table 5: Reactivity Test

Replacement	Observation	Soluble	Precipitate
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Reaction Product			
NH₄NO₃+AgCl	White, milky, cloudy precipitate	Not soluble	Milky Precipitate
NH₃H₂O+NaCl	Ammonia smell	Soluble	Gas
NH₄OH+KCI	Very faint ammonia smell	Soluble	Gas
NH₄OH+MgCl₂	No reaction or color change	Soluble	None
(NH ₄) ₂ SO ₄ +FeCl ₂	No reaction but very light yellow color	Soluble	None

Table 6: Quantitative Analysis Results

Total Mass(g)	Filter Paper Mass(g)	Mass of AgCl
1.191	0.678	0.513
1.237	0.668	0.569
1.197	0.679	0.518

Average Mass of AgCl: 0.533g AgCl

Figure 1: Quantitative Analysis Calculations

$$\begin{split} \text{NH}_4\text{Cl}_{(aq)} + \text{AgNO}_{3(aq)} &\to \text{AgCl}_{(s)} + \text{NH}_4\text{NO}_{3(aq)} \\ \text{N = 14.01g x 1 = 14.01g} \\ \text{H = 1.008g x 4 = 4.032g} \\ \text{Cl = 35.45g x 1 = } \frac{35.45\text{g}}{53.49\frac{g}{mol}} \text{ NH}_4\text{Cl} \end{split}$$

$$\frac{\textit{amount of compound }(g)}{\textit{molar mass of compound }\left(\frac{g}{\textit{mol}}\right)} = \frac{0.2g~\text{NH}_4\text{Cl}}{53.49\frac{g}{\textit{mol}}\text{NH}_4\text{Cl}} =$$

0.00374 mol NH4Cl

$$\begin{aligned} \textit{mole ratio of reaction} &= \frac{1 \, \textit{mol of NH}_4 \textit{Cl}}{1 \, \textit{mol of AgCl}} \\ &= \frac{0.00374 \, \textit{mol NH}_4 \textit{Cl}}{0.00374 \, \textit{mol AgCl}} \end{aligned}$$

Ag = 107.87g x 1 = 107.87g
Cl = 34.453g x 1 =
$$\frac{34.453g}{142.32 \frac{g}{mol}}$$
 AgCl

molar mass of AgCl x mol of AgCl = Theoretical Yield in grams

$$142.32 \frac{g}{mol}$$
 AgCl x 0.00374 mol AgCl = **0.532g AgCl = Theoretical Yield**

$$\begin{split} \textit{Actual mole of AgCl} &= \frac{\textit{average weight of AgCl}}{\textit{molar mass of AgCl}} \\ &= \frac{0.533g~\text{AgCl}}{142.32\frac{\textit{g}}{\textit{mol}}~\text{AgCl}} = \textbf{0.00375~\textit{mol AgCl}} \\ \textit{Actual mole ratio} &= \frac{0.00374~\textit{mol NH}_{4}\text{Cl}}{0.00375~\textit{mol AgCl}} = \textbf{0.997~\textit{mole ratio}} \end{split}$$

$$Percent \ error = \left| \frac{Theoretical \ Value - Experimental \ Value}{Theoretical \ Value} \right| \ x \ 100$$

$$= \left| \frac{1 \ mole \ ratio - 0.997 \ mole \ ratio}{1 \ mole \ ratio} \right| \ x \ 100 = \ \mathbf{0.3\%} \ error$$

Figure 2: Synthesizing Ammonium Chloride Calculations

$$(NH_4)_2CO_3 + 2 HCl \rightarrow CO_2 + H_2O + 2 NH_4Cl$$

$$\frac{\textit{amount of compound }(g)}{\textit{molar mass of compound }\left(\frac{g}{\textit{mol}}\right)} = \frac{5.0g \; \text{NH}_4\text{Cl}}{53.49 \frac{g}{\textit{mol}} \; \text{NH}_4\text{Cl}} =$$

0.0935 mol NH4Cl

mole ratio of reaction =
$$\frac{1 \text{ mol of (NH4)2CO3}}{2 \text{ mol of NH4Cl}}$$
$$= \frac{0.0468 \text{ mol (NH4)2CO3}}{0.0935 \text{ mol NH4Cl}}$$

molar mass of (NH₄)₂CO₃ x mol of (NH₄)₂CO₃ = Theoretical Yield in grams $96.086 \frac{g}{mol} \text{ (NH₄)}_2\text{CO}_3 \times 0.0468 \text{ mol (NH₄)}_2\text{CO}_3 = 4.50 \text{g (NH₄)}_2\text{CO}_3$