Experiment 3: [http://genchemlab.wordpress.com/3-empirical-formula/](http://genchemlab.wordpress.com/3-empirical-formula/)

**Objectives**
- To learn how to do a gravity filtration.
- To determine the percent composition of a compound.
- To determine the empirical formula of a compound.
- To learn about hydrated compounds.

**In the Lab**
- Students will work in pairs.

**Waste**
- The filtrate (liquid) should be disposed of in the aqueous waste container.
- The dried copper salt should be poured into the container marked “Copper Waste.”
- The copper should be disposed of in the bucket marked “Copper Waste.”
- Used aluminum should be disposed of in the waste container marked “Aluminum Waste.”

**Safety**
- Students should wear gloves when working with the copper salt solution.
- Take care in handling solid reagents to avoid inhalation and eye exposure. Always use a brush to clean the balance after each use.
- Always wash your hands before leaving the laboratory.
The empirical formula of a compound can be determined based on experimental data. In order to do this, the percent composition by mass of the different components must be determined in the laboratory. Using these percentages, one can determine the empirical formula for the compound. The resulting formula may support other experimental results regarding the identity of a substance or it may disprove the hypothesized identity of the substance. Mass percent is calculated using the following formula:

$$\text{mass \% of A} = \frac{\text{mass of A}}{\text{total mass of substance}} \times 100$$

where A is one component of the substance. Because mass percent is an intensive property (independent of amount), we can compare mass percent values from multiple experiments if they are done on portions of a common sample. In this experiment, we will determine the mass percent of copper and the mass percent of water in a copper salt which has the general formula $\text{Cu}_x\text{Cl}_y\cdot z\text{H}_2\text{O}$. Once we determine the percents of water and copper, we can determine the percent of chlorine by subtracting those values from 100.

**Mass Percent of $\text{H}_2\text{O}$**

This salt is a hydrate, which means it has a specific number of water molecules associated with it. By drying a sample of this salt, we can determine the mass of water lost and determine the mass percent of water in the compound. The mass of any water molecule(s) must be included when calculating the molar mass of a hydrate, because the mass of these waters are included in any measurements taken on an analytical balance.

**Mass Percent of Copper**

To determine the amount of copper in this salt, we must perform a chemical reaction that results in the formation of elemental copper. As shown in the reaction, the copper salt will react with aluminum to form elemental copper and aluminum chloride. The hydrate is not written as part of the reaction because the copper salt is dissolved in water, indicated by (aq) for aqueous.

$$\text{Al(s)} + \text{Cu}_x\text{Cl}_y(aq) \rightarrow \text{Cu(s)} + \text{AlCl}_3(aq) \quad \text{[unbalanced]}$$

While this reaction produces copper, the solid copper must be separated from the aqueous aluminum chloride solution and dried. If we do not isolate and dry the copper before determining its mass, the measured value would also include some water and/or $\text{AlCl}_3$ which would affect the results of the experiment.

**Empirical Formula**

Once the experiment(s) have been done to determine the mass percentages, it only takes a few calculations to determine the empirical formula. Remember that an empirical formula is not the same as a molecular formula because the subscripts have been reduced to the smallest whole number ratios. The empirical formula can be determined from the mass percentages based on the following steps:

1. Assume a 100 gram sample. Any mass can be assumed, but 100 grams is the most convenient.

2. Change percentages of a component to the mass of a component. If we assume 100 g, then this is just a change of units (i.e., 34% to 34 g).

3. Convert mass of a component to moles.

4. Divide all of the moles by the smallest value to determine the relative number of moles of each component.

Examples in books tend to come out with nice, whole number ratios because they were written that way. In the lab, you may not get nice, whole number ratios and you may hear the phrase “round generously.” While you are learning about rounding to the correct number of significant figures, you need to remember that experimental results for this experiment may not be as neat as the examples given in textbooks. In order to complete the data analysis, we have to work through this to focus on the concepts rather than getting caught up on whether or not to round 2.4 down to 2 or not. The emphasis in this, and in many other experiments, is on understanding the concepts rather than getting the right or wrong answer.
Experiment 3 • Empirical Formula of a Copper Salt

Safety Note
Do not wear gloves out of the laboratory. Any chemicals on your gloves will be spread to whatever you touch such as the water fountain or the bathroom door. Anyone coming along behind you could then be unknowingly exposed to a chemical. You could also put yourself in contact with chemicals by touching your face when wearing gloves even if the gloves appear to be clean.

Even if you have not yet started working in the lab, do not wear gloves out of the laboratory for any reason. Imagine walking into a restaurant and seeing an employee wearing gloves while taking out the trash. Then that same employee, still wearing gloves, prepares your food. How do you know they aren’t wearing the same pair of gloves? Would you want them preparing your food?

Since it is not always possible to tell whether your gloves have been in contact with a chemical, it is assumed that all gloves have been in contact with a chemical once you put them on your hands.

Materials
- copper salt
- crucible with cover
- crucible tongs
- 100 mL beaker
- wash bottle
- 6 M HCl
- aluminum wire
- 11-cm filter paper
- funnel
- 125 mL Erlenmeyer flask
- watch glass
- spatula

Procedure

Percent of H₂O
1. Obtain 0.4–0.5 g of the copper salt and record the exact mass. See Chapter 3 for instructions on how to use an electronic balance.
2. Use a permanent marker to write your initials on the crucible.
3. Record the exact mass of the crucible and cover.
4. Place the uncovered sample in the crucible and place on a tile in the oven until the sample is completely brown (approximately 30 minutes).
5. Use crucible tongs to remove the crucible from the oven. Immediately, put the lid on the crucible.
6. Allow the sample and crucible to cool for approximately 5–10 minutes and then record the mass. The data collected from this part will be used to determine the mass percent of water in the sample.
7. Pour only the dried copper salt into the container marked “Used Copper Salt.” Once it has been rehydrated, it can be reused.

Percent of Cu
1. Obtain another 0.4–0.5 g sample of the copper salt and record the exact mass. If you get too much, the experiment will take too long to complete during your lab session.
2. Place the copper salt sample in a 100 mL beaker. Use your wash bottle with distilled water to rinse the weighing boat into the beaker to remove all of the copper salt.
3. Add more distilled water to a volume of approximately 20 mL (the exact volume does not matter).
4. Add approximately 1 mL (20 drops) of 6 M HCl (the exact volume does not matter) to the beaker.

5. Swirl the solution until the copper compound is dissolved; you should have a blue, transparent solution.

6. Obtain an aluminum wire and place it in the beaker with the copper salt solution. Swirl the beaker to remove the forming copper from the wire.

7. Continue until the blue color is gone from the solution (~15 minutes). Place beaker on a white piece of paper to detect the presence or lack of color. Solution may be slightly cloudy, but should not appear blue at all.

8. Remove the aluminum wire from the solution using a glass stir rod. Rinse the wire with distilled water to remove all of the copper from the aluminum wire and collect rinsings in the beaker. Try not to lose any of the copper.

9. Obtain a piece of 11 cm filter paper and use a pencil to write your name on it.

10. Following Figure 3.1, set up your filter paper and place it in the funnel. Place the funnel in the 125 mL Erlenmeyer flask.

11. Completely wet the filter paper with distilled water.

12. Filter the copper from the solution. It may be necessary to pour the solution into the funnel in portions so that you do not overfill the filter paper. Use your wash bottle with distilled water to remove all of the copper from the beaker.

13. Rinse copper with three portions of water. When rinsing a sample, pour a small amount of the liquid over the sample (usually less than 5 mL). Allow the liquid to filter through the sample and then repeat, as needed.

14. Dispose of the filtrate (the liquid) in the aqueous waste container. Allow the filtrant (the solid) to sit for approximately 5 minutes to drain as much water as possible.

15. Use a permanent marker to put your initials on the watch glass.

16. Determine the mass of the watch glass.

17. Open filter paper, place it on the watch glass, and lay it in the oven.

18. After 5 minutes, remove filter paper from oven and scrape sample onto the watch glass. Use spatula to spread out the copper. Place watch glass in oven. Leave sample in oven to dry for at least 20 minutes.

19. Remove sample from oven, allow it to cool for 5–10 minutes, and record its mass. Check that you have your sample.

20. Dispose of the copper and filter paper in the container labeled “Copper Waste.”

21. Dispose of the clean, dry aluminum wire in the container labeled “Used Aluminum.”
Data Analysis

1. What is the mass of the dried copper salt?

2. What mass of water was lost?

3. What is the mass percent of water present in the copper salt?

4. What is the mass of copper present in the copper salt?

5. What is the mass percent of copper present in the copper salt?

6. What is the mass percent of chloride present in the copper salt?

7–9. Use the mass percentages you found and assume a 100 g sample to determine the number of moles of copper, chlorine, and water.

10. What is the empirical formula of Cu\text{_{x}}Cl\text{_{y}} \cdot z\text{H}_{2}\text{O}?
    Whole number values only for x, y, and z.
Expt. 3 • Empirical Formula of a Copper Salt