EXPERIMENTAL DETERMINATION OF AN EMPIRICAL FORMULA

Prepared by M.L. Holland, PhD, Foothill College

Purpose of the Experiment

- Understand how to determine an empirical formula from experimental data
- Practice measuring and recording quantities of chemicals correctly
- Become familiar with the concept of an excess reactant

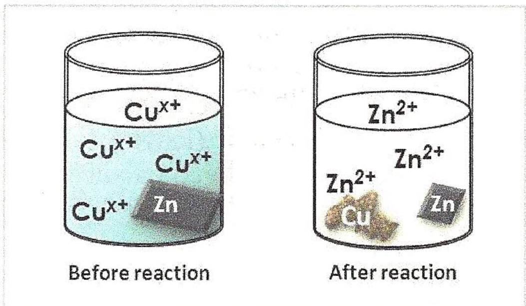
PreLab Preparation:
Complete the online prelab assignment and Lab Procedure Outline as your instructor assigned

Background Information:
Empirical Formulas: As we learned in the previous lab, chemical formulas tell us which elements are present in a compound and how many atoms of each element are in the compound. Chemical formulas also tell us the number of moles of each element that are present in one mole of the compound. Another way of stating this is to say the formulas tell us the mole ratios of the elements in the compound. The compound sodium nitrate, $NaNO_3$, has the mole ratio of 1 mol Na to 1 mol N to 3 mol O. Chemists must often determine the formula of an unknown compound by experiments in which the mass of each element in a sample of an unknown is measured. The number of moles of each element in the sample can then be calculated and used to find the lowest whole number ratio of the elements. Formulas determined by this approach are called *empirical* formulas.

The word *empirical* means *determined by experiment* and empirical formulas represent the *lowest whole number ratio* of the elements in the compound. For ionic compounds, the empirical formula will be the same as the "molecular" form of their formula, but molecular compounds may have different empirical and molecular formulas. For example, the molecular formula for the sugar glucose is $C_6H_{12}O_6$, but the empirical formula is CH_2O (the lowest whole number ratio of the subscripts 6, 12 and 6).

In this lab, we will be determining the empirical formula for an ionic compound composed of copper and chloride ions. Copper is a transition metal that can form more than one ion, so we cannot determine the correct formula for our compound just by knowing it contains copper and chloride ions, because we do not know the charge on the copper ion in the compound. We can, however, perform an experiment to determine the correct formula.

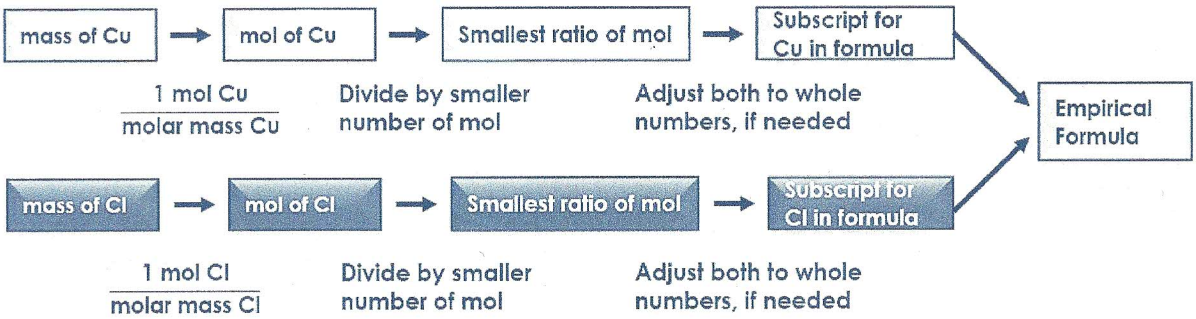
We will use a chemical reaction to replace the copper in our compound (Cu_xCl_y) with zinc and then recover the pure copper as a solid. The reaction equation (not balanced) is shown below:



$$\_\_\,Cu_xCl_y\,(aq) + \_\_\,Zn(s) \rightarrow \_\_\,Cu(s) + \_\_\,ZnCl_2\,(aq)$$

Notice that our copper chloride compound is dissolved in water (aq). We will measure a precise volume of a stock copper chloride solution and calculate the mass of our copper chloride sample using the given concentration of the stock solution. We will add a piece of solid zinc to our copper chloride solution and a single displacement reaction will occur. The zinc will become ionized by transferring electrons to the copper ions and go into solution, displacing the copper ions. The displaced copper with the transferred electron(s) will be deposited on the surface of the zinc as solid copper. There will be zinc metal left over after the reaction, because we are deliberately using an excess of zinc to ensure that all of the copper ions are removed from the solution. Note that there are also Cl^- ions in our beaker solution both before and after the reaction, but they are not shown in the figure because they are not participants in the reaction. They just remain in solution.

After we have reacted all of the copper ions in solution and formed solid copper, we will recover the copper and weigh our rinsed and dried copper. The data values we need to calculate the empirical formula are the mass of Cu_xCl_y in our sample of stock solution, and the mass of the copper we recover. We will determine the mass of chlorine in our sample by subtracting the mass of the copper recovered from the mass of Cu_xCl_y. Once we have the mass of both the Cu and the Cl in our sample, we calculate the empirical formula using the following process:



By the end of this lab, you should be familiar with the concept of using a chemical reaction to obtain data that will allow the calculation of a simple unknown empirical formula, record mass and volume measurements with the correct number of digits and calculate the empirical formula of a compound from experimental data.

Equipment and Reagents:

Cu_xCl_y stock solution (~0.08_ _g/mL)	wire gauze	glass stir rod/rubber tip
zinc pieces (~1.5 g total mass)	tweezers	top loading balance
10% HCl in a dropper bottle	microspatula	100 mL graduated cylinder
hot plate	2 – 150 mL beakers	beaker tongs

Safety: Always wear your safety goggles while in the lab room. Avoid skin contact any of the solutions used in this lab and wash your hands before leaving lab. The Cu_xCl_y stock solution can be an irritant and the hydrochloric acid is corrosive. Keep balance areas clean and carefully wipe up any spills with a damp paper towel.

Procedure: (Work with a partner; each partner should do all calculations individually)

Performing the Single Displacement Reaction

8. Get a hot plate and set it to heat setting of 5 (medium heat). If there is a stir knob, set it to zero.
9. Weigh a clean, dry 150 mL beaker and record the mass on your data sheet (all digits)
10. Take your 100 mL graduated cylinder to the bench with the Cu_xCl_y stock solution. Use a large plastic pipet to transfer approximately 25 mL of the solution into your 100 mL graduated cylinder. Record the measured volume of solution (1 decimal place)and the precise concentration of the stock solution from its label on your data sheet.
11. Carefully pour the stock solution into a 150 mL beaker, then add about 5 ml of water to the cylinder, swirl it and pour the rinse water into the 150 mL beaker. The purpose of the rinse is to ensure we transfer every drop of blue Cu_xCl_y stock solution into our reaction beaker.
12. Obtain a zinc piece and weigh it by taring a piece of weighing paper, putting the zinc on the weighing paper and recording the mass of the zinc (record all balance digits). Your zinc should weigh at least 1.5 g, but no more than 2.5 g. You may need 2 pieces of zinc to be in this mass range.
13. Carefully slide the zinc into your 150 mL reaction beaker containing the Cu_xCl_y stock solution.
14. Use the rubber end of your glass stir rod to gently remove the dark solid that appears on the surface of the zinc. The reaction occurs at the surface of the zinc, so we want to keep the zinc surface clear so the reaction can go to completion. Keep removing the dark solid (take turns with your partner) until the blue color of the solution is completely gone. You can place your beaker on a white surface to check the color of the solution.
15. Once the color is gone, add 5-10 drops of the 10% HCl solution and stir gently, but thoroughly. The dark solid particles should now be more copper-colored.
16. Remove the excess zinc from the beaker using tweezers and place on a piece of paper towel. Using your small metal spatula, scrape off any copper on the surface of the zinc and return the copper to the reaction beaker. Dry the zinc well and weigh it on the same balance. The zinc can then be discarded into the labeled jug.

Recovering the Copper

11. Carefully decant most of the liquid from your mixture into your waste beaker.
12. Add 20 mL deionized water to the beaker and swirl to mix. Decant most of the liquid into your waste beaker.
13. Repeat Step 2 one more time to thoroughly rinse any $ZnCl_2$ out of the copper. Decant as much water as you can into the waste beaker, using your stir rod to keep the copper in the beaker.
14. Place the beaker on your hot plate and heat for 15 min. You may use your small metal spatula or the glass end of your stir rod to gently break up any clumps of copper. Heat until your copper appears dry and crumbly.
15. Remove beaker from hot plate and let cool for ~ 5 min. Weigh the beaker and record the mass as beaker and copper after 1st heating.
16. Place the beaker on your hot plate again and heat for an additional 5 min.
17. Remove beaker from hot plate and let cool for ~ 5 min. Weigh the beaker and record the mass as beaker and copper after 2nd heating.
18. Use the **lower** of the two mass values after heating for your calculations.
19. The copper should be discarded into the labeled jug.

Data, Calculations and Discussion

Record all data in the indicated spaces in the Data Table. Complete all calculations on the table and answer the discussion questions with your partner before leaving. Follow your instructor's directions for turning in the Data, Calculations and Discussion Questions pages.

Name____________________ **Partner**____________________ **#**______

Data and Calculations

Data	**Value (units)**
Concentration of Stock Cu_xCl_y Solution from label on bottle (g/mL)	
Volume of Stock Cu_xCl_y used (to 1 decimal place)	
Mass of empty beaker (all balance digits)	
Mass of beaker and copper (after 1st heating; all digits)	
Mass of beaker and copper (after 2nd heating; all digits)	
Starting mass of zinc	
Mass of zinc recovered (final mass of zinc)	
Calculations (Show set-ups)	
Mass of Cu_xCl_y in your stock solution sample: (Volume of stock Cu_xCl_y used x concentration of stock Cu_xCl_y solution)	
Mass of copper recovered (mass of beaker and copper (*<u>lowest mass after heating</u>*)– mass of empty beaker)	
Mass of chlorine in your stock solution sample (Mass of Cu_xCl_y in your stock solution sample – mass of copper recovered):	
Determination of Empirical Formula (Show set-ups) Using the masses of Cu and Cl in our sample, we determine the empirical formula by converting the masses to moles and finding the lowest whole number ratio of the numbers of moles. (See page 2)	
Convert masses of copper and chlorine to mol: $\text{g Cu x} \frac{\text{1 mol Cu}}{\text{molar mass of Cu}} = \text{mol Cu}$ $\text{g Cl x} \frac{\text{1 mol Cl}}{\text{molar mass of Cl}} = \text{mol Cl}$	mol Cu: ----------------- mol Cl: -----------------
Divide by smaller number of mol: *(show work, including values before rounding)* mol Cu/smaller number of mol mol Cl/smaller number of mol	<u>Mole ratio</u> Cu: Cl:
Empirical formula: For this lab, we will round the results for mol Cu and Cl to the nearest whole number. The rounded whole numbers are the subscripts in the empirical formula. **Write your empirical formula ⇒**	**Cu Cl**

Discussion Questions:

1. Write **your** balanced equation for today's reaction, using **your** empirical formula:

   a. Calculate the mass and then moles of Zinc reacted as follows:
   Mass of Zn reacted = starting mass of Zn – final mass of Zn **Mass of Zn:**_______

   Mol of Zn reacted = Mass of Zn reacted $\times \frac{1 \text{ mol}}{\text{molar mass of Zn}}$ **Mol of Zn:**_______

   b. Compare your mol of Zn reacted to your mol of Cu from your data sheet. Are they approximately the same?
   Mol of copper recovered (from data sheet)_______ vs. mol of Zn reacted ______

2. Should the moles of Zn reacted and copper recovered be approximately the same based on your balanced equation above? Explain why or why not?

3. Should everyone in the class obtain the same formula for our Cu_xCl_y if they performed the experiment correctly? Briefly explain your answer.

4. Consider the potential effect of the errors below on the determination of the empirical formula.
   a. A student stirred their reaction vigorously, which caused some small pieces of zinc to break off the bigger zinc strip. These small pieces of zinc remained in the beaker with the copper after the solution was decanted. What effect would this error have on the **calculated moles of copper and chlorine**? Explain your answer. (*Hint: the effect may be different for Cu vs. Cl*)

   b. A student added double the amount of zinc to their reaction mixture. What effect would this error have on the **calculated moles of copper and chlorine**? Consider how the moles of copper and chlorine are calculated when you answer this question **AND** whether there is any copper left in your solution to react with the extra zinc.

5. A similar experiment to today's lab was performed to determine the empirical formula of a compound containing iron and chlorine. The data obtained were:
   Mass of Fe_xCl_y sample: 3.458 g Mass of recovered iron: 1.190 g

   a. What is the empirical formula for this compound?

   b. What is the mass percent iron in this compound based on the formula?