CH 221 Fall 2024:
"Empirical Formula" *(in class)* ***Lab - Instructions***

Note: **This is the lab for section 01 and H1 of CH 221 only.**

- *If you are taking section W1 of CH 221*, please use this link:
  http://mhchem.org/s/4b.htm

Step One:

Get a printed copy of this lab! You will need a printed (hard copy) version of pages Ia-4-2 through Ia-4-11 to complete this lab. If you do not turn in a printed copy of the lab, there will be a 2-point deduction.

Step Two:

Bring the printed copy of the lab with you on Monday, October 14 (section 01) *or* **Wednesday, October 16 (section H1).** During lab in room AC 2507, you will use these sheets (with the valuable instructions!) to gather data, all of which will be recorded in the printed pages below.

Step Three:

Complete the lab work and calculations on your own, then **turn it in** (pages Ia-4-7 through Ia-4-11 *only* to avoid a point penalty) **at the beginning of recitation to the instructor on Monday, October 21 (section 01)** *or* **Wednesday, October 23 (section H1.)** The graded lab will be returned to you the following week during recitation.

If you have any questions regarding this assignment, please email (mike.russell@mhcc.edu) the instructor! Good luck on this assignment!

Empirical Formula

One of the fundamental statements of the atomic theory is that elements combine in simple whole number ratios. This observation gives support to the theory of atoms, since one would expect whole atoms to combine. Furthermore, it is observed the combining ratio for a given compound is constant regardless of the origin of the pure substance. This is known as the **Law of constant composition**. The mass contribution of each atom in a compound is a function of the number of atoms in the simplest formula and the relative mass of each atom. The mass contribution is usually referred to as the **percent composition** of a compound.

The **empirical formula** represents the smallest whole number ratio of atoms in a compound. The **molecular formula** represents the actual number of atoms in a compound. The molecular formula may be the same as the empirical formula or it may be a multiple of the empirical formula. For example, hydrogen peroxide has a molecular formula of H_2O_2 and an empirical formula of HO, while water has a molecular formula of H_2O and an empirical formula of H_2O. *No fractions should be present in empirical or molecular formulas!*

Because atoms combine in a definite ratio, the mass composition or **percent by mass** of a compound is fixed. You can determine the mass contribution of each element in a compound by the number of atoms in the simplest formula and the relative mass of each atom. This is typically referred to as the **percent composition** by mass of a compound. For example, $CuCO_3$ is always **38.847%** oxygen by mass:

$$\% \text{ Oxygen} = \left(\frac{3 \text{ oxygen atoms x } 15.999 \text{ amu per oxygen}}{123.554 \text{ amu total molar mass for } CuCO_3} \right) \text{ x } 100\%$$

In this experiment you will determine the empirical formula of a hydrated compound of copper and chloride. You will first remove the water from the compound by heating the sample. Any mass lost is water. To determine the mass of copper in the compound, a simple exchange reaction with zinc is performed. Zinc is referred to as an active metal; in contact with a solution containing copper ions, the zinc metal will react to convert the copper ions into copper metal (zinc is transformed into zinc chloride.) As long as excess zinc is added, all of the copper ion should be removed as copper metal, which is easily massed. Any undetermined, remaining mass is chloride.

Once the mass of each component of the compound is determined (water, copper, chloride), you can calculate their corresponding moles and determine the empirical formula.

Because you are novice chemists, your experimental data will not be perfect. By following significant digit rules and using reasonable analytical deduction, you should be able to determine the formula of the hydrate. You can discuss your rounding and sources of error in your lab report.

In addition, you will be able to calculate the **theoretical yield** of copper based on the amount of zinc reacted. Comparing the **actual yield** of copper recovered to the theoretical yield, you can determine a **percent yield** of copper.

Operating a Bunsen Burner

Bunsen burners rely on the combustion of natural gas. An optimal mixture of gas (methane, CH_4) and air (oxygen) will produce a flame with an obvious blue (oxidizing) cone. **To set up your Bunsen burner:**

1. Attach one end of the rubber tubing to the sidearm at the base of the Bunsen burner and attach the other end to a gas outlet. (Be certain that the outlet is labeled "gas".)
2. Adjust the air (oxygen) intake to halfway (see picture below).
3. Adjust your ring stand to the correct height. The ideal flame should be only 2-3 cm above the burner. The ring and stand are metal and will be too hot to adjust once you begin heating. If you are unsure how to properly set up your ring stand, politely ask your instructor for assistance.
4. Make sure you can obtain a spark from the striker before proceeding.

To light your Bunsen burner you must do two things <u>simultaneously</u>:

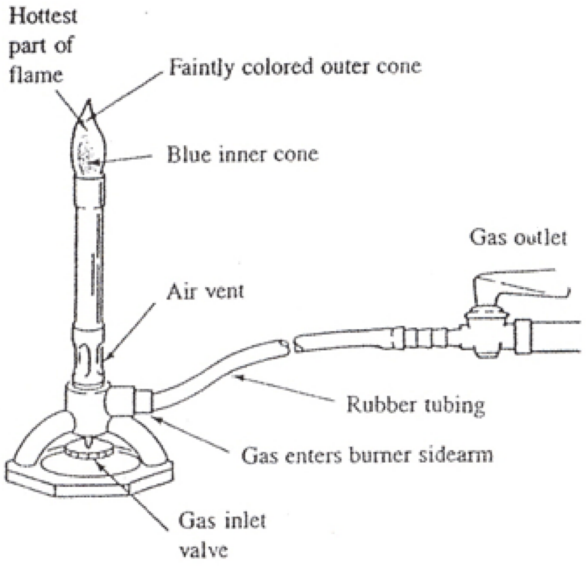
1. Open the gas inlet valve on the burner about halfway (see picture below).
2. As you open the gas outlet, light the burner by bringing the striker from the side to the top.

Do not leave the gas outlet open without the burner lit. It is unsafe to allow natural gas to enter the lab. Always check that the gas outlet is turned off when you are not using the burner. If you are unsure how to light the burner, please patiently ask your instructor for assistance.

To adjust your Bunsen burner:

Adjust the air (oxygen) intake until the flame becomes two concentric cones about 2-3 cm above the burner. The outer cone will be only faintly (dark blue) colored, but the inner cone will be a light blue color. The hottest part of the flame is at the tip of the inner light blue cone. If the flame is luminous and yellow or orange, but not blue, the air vent is not well adjusted. To adjust the height of the flame, adjust the gas flow at the gas outlet or at the gas inlet at the bottom of the Bunsen burner. Proper burner adjustment is crucial for good results. If you are unsure how to adjust the flame, please graciously ask your instructor for help.

Metal and ceramics hold heat well. Be careful when heating metal and ceramics as they will stay hot for quite a while after you turn the burner off. If unsure, remember that patience is a virtue, and wait longer for it to cool.



PROCEDURE: Part A: Dehydration of the copper compound

1. Record the mass of a clean, dry small crucible (no lid) in the "Data" section, below. ***All*** **measurements obtained in this lab should be to the nearest milligram (0.001g).**

2. Place approximately one gram (1.0 to 1.2 g) of unknown hydrated copper chloride in the crucible. Break up any sizable crystals with your spatula by pressing against the side of the crucible. Record the mass of the crucible (no lid) and sample to the nearest milligram.

3. Place the uncovered crucible on a clay triangle supported by a ring stand. Light your Bunsen burner away from the crucible, adjust the flame as described in the introduction, and ***gently*** heat the crucible as you move the burner back and forth judiciously. Flame should be small to avoid "popping" of sample. *If you overheat your sample*, it will turn into a black nasty liquid, and you must start over... so **heat gently!!!**

4. You should notice the crystals change color as they are heated. Record observations. Why do the crystals change color when heated? Continue to slowly heat the sample until all the crystals are brown. After about 5 minutes, carefully stir sample with a glass stir rod to check color. Once the entire sample is brown, gently heat for two additional minutes.

5. Turn off the burner. Cover the crucible as it cools to prevent the re-absorption of water vapor. Cool the crucible for about 10 minutes. *Caution:* The crucible is ceramic and retains heat. The crucible can severely burn you if you try to touch it before it is cool. Patience is a virtue!

6. After 10 minutes, remove the cover and slowly roll the brown crystals around the crucible. If there is any evidence of green crystals, repeat the heating and cooling process... but if all the crystals appear brown and the crucible is cool, **record the mass of the crucible (no lid) and dehydrated sample**.

Part B: Exchange reaction between the copper compound and zinc

1. Transfer the brown crystals to a small 250 mL beaker. Rinse the crucible with two 5-7 mL portions of deionized H_2O, adding each rinse to the beaker. Swirl the beaker gently to dissolve the crystals and record your observations. Why did the color change?

2. Obtain a piece of clean zinc and record its mass to the nearest 0.001 g. You need at least 0.5-0.8g of zinc; more is fine. Gently slide the piece of zinc into the beaker so that it is submerged in the copper chloride solution. Be careful not to splash. Add ~5 mL of water if your volume is too low.

3. Stir the solution with a glass rod so that as copper forms, it does not adhere to the zinc. Record observations. Allow the reaction to continue until all blue and green color has disappeared from the solution. The solution might have an unattractive grey hue, but no tint of green should remain.

4. Add 10 drops of 10% HCl to the solution and stir thoroughly. This will dissolve any insoluble zinc salts formed and clear up the solution if cloudy.

5. Carefully remove the unreacted zinc metal from the solution using tongs. Inspect the zinc for any adhering copper. Use a wash bottle of deionized water and a rubber policeman to scrape and clean the copper off the zinc into the beaker. Dry the remaining zinc on a paper towel. Record the mass of the dry zinc. (Note: this

must be less than your starting mass, right?) Place the zinc in the waste container when this step is complete.

Part C: Cleaning the copper:

1. Set up a Buchner funnel suction filtration apparatus with a moistened piece of filter paper. Attach the rubber hose to the "VAC" outlet, and only turn the vacuum to a 45 degree angle initially to prevent losing copper.

2. With light suction, carefully decant (pour off) the solution over the copper into the funnel. It is okay if some of the copper is transferred to the funnel.

3. Wash the copper solid in the beaker with about 10 mL of deionized water. Stir thoroughly, allow the copper to settle and carefully decant the wash water into the funnel. Break up any large chunks of copper with your glass stir rod. Repeat with a second 10 mL portion of deionized water.

4. Transfer the copper to the funnel using a small amount of deionized water. Use your wash bottle and rubber policeman to facilitate the transfer all of the copper to the funnel. Rinse any copper adhering to the rubber policeman into the funnel. All of the copper must be transferred to the funnel.

5. Turn off the suction. Add 10 mL of methanol to the funnel. After one minute, turn on the suction (slow at first, then to a roughly 45 degree angle.) Methanol evaporates faster than water and will enhance the drying process.

6. Draw air through the funnel for about 3-5 minutes. Meanwhile, record the mass of a clean, dry watch glass.

7. Transfer the dry copper to the massed watch glass. The transfer must be quantitative; scrape any copper that adheres to the paper on to the watch glass with your spatula or rubber policeman. If the copper is still damp, dry under a heat lamp for 5 minutes or press with a dry piece of filter paper. Allow the sample and watch glass to cool. Record the mass of the copper to the nearest 0.001 g. If you have more than 0.50 grams of copper, your sample is probably wet. It is recommended that you dry it under a heat lamp and take a new measurement.

8. Clean up! Dispose of the liquid methanol waste from the suction filtration apparatus into the appropriate waste bottle. Discard the copper in the garbage can unless directed otherwise by the instructor.

9. Complete the worksheets below using the data obtained in lab.

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Empirical Formula Worksheet

Name:

Complete the worksheets below and turn in on the due date

Lab Partner(s):

All mass measurements should be made to the nearest milligram (0.001 g).

Mass of clean, dry, small crucible (g): _____

Mass of crucible and copper chloride sample (g): _____

Mass of unheated copper chloride sample (g): _____

Color of copper chloride sample before heating: _____

Mass of crucible and dehydrated copper chloride sample (g): _____

Mass of dehydrated copper chloride sample (g): _____

Mass of water lost upon heating (g): _____

Color of dehydrated copper chloride: _____

Color of dehydrated copper chloride sample when dissolved in water: _____

Mass of zinc sample before reaction (g): _____

Observations as zinc is added to the copper solution:

Mass of dry zinc after reacting with copper (g): _____

Mass of zinc that reacted with copper (g): _____

Mass of clean, dry watch glass (g): _____

Mass of dried copper and watch glass (g): _____

Mass of dried copper (g): _____

Color of dried copper: _____

Mass of chloride (g): *(dehydrated copper chloride - dried copper)* _____

Calculations Worksheet *for the* **Empirical Formula** *Lab*
Do not wait until the last minute to start these calculations!

1. Calculate the **mass (grams) of water** lost upon heating the copper chloride sample. Calculate the **moles of water lost** upon heating the hydrated copper compound

2. Calculate the **mass (grams) of copper** collected at the very end of lab; this is your **actual yield** of copper (g). Determine the **mass (grams) of chloride lost** in your sample (mass of dehydrated copper sample – mass of copper collected after filtration = mass of chloride.) Convert mass (grams) of copper into **moles of copper**; also convert mass (grams) of chloride into **moles of chloride.**

3. Use the moles of water, moles of copper and moles of chloride to **find the empirical formula of the hydrated copper chloride**. Round to whole numbers when determining the empirical formula.

4. Use the masses of water, copper, chloride, and the original hydrated copper chloride sample to **find the percent copper, percent chloride and percent of water in the original hydrated copper chloride sample**.

5. Show how to calculate the **mass (g) of zinc reacted** (i.e. the initial weight of Zn minus the final weight of Zn after the reaction was complete). Calculate the **theoretical yield of copper** using the formula:

   Theoretical yield (grams) of Cu = (grams of zinc reacted in the reaction) * 0.9720

6. Calculate the **percent yield** of copper. %yield = (actual yield / theoretical yield) x100%. **Comment** on why the percent yield might be greater than 100% in this lab.

POSTLAB QUESTIONS:

1. The *limiting reactant* (also known as the limiting reagent) is defined as the starting substance which is totally consumed in a reaction; the *excess reactant* is a starting material which is still present at the end of a reaction. Which of the reactants was the limiting reactant and which was the excess reactant? (the reactants in this reaction were zinc metal and the unknown copper chloride.) *Briefly* explain your answer.

2. Explain the color changes in part A (from blue to brown and back to blue) *and* in part B (from blue to clear).

   Part A:

   Part B:

3. **Explain** the effect each of the following would have on the experimentally determined %Cu. Use the terms **increase**, **decrease** or have **no effect** to describe the effect on the %Cu.
   a. Some solution splashed onto the bench when the zinc was plopped into the beaker.

   b. The student removed the zinc before the blue color disappeared from the solution.

   c. The student did not completely dry the Cu before the final weighing

4. Your final mass of zinc was less than your initial. What happened to the zinc? What did it become?

5. Determine the %Cl by mass value if the sample was pure anhydrous copper(II) chloride. *Hint:* do not use your data for this question; use the formula... what *is* the formula for copper(II) chloride?

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