# *The Copper Cycle*

In this experiment, you will carry out a series of reactions to transform copper into various forms and learn more about the three main types of chemical reactions: precipitation, acid-base neutralization, and redox reactions. You will also determine the efficiency of the overall transformation cycle by calculating the percentage of starting material recovered at the end of the experiment.

The chemistry behind each step is described in the Background section of this experiment. The procedures required to carry out each step are described below.

## ****Lab Notebook****

You should include one table that contains the mass of copper at the beginning and end of the experiment along with % of copper recovered. This table should include:

- Mass of copper at the start of experiment (in Part I)
- Mass of copper + evaporating dish (from Part V)
- Mass of empty evaporating dish (from Part V)
- Mass of copper recovered (from Part V)
- Percent of copper recovered

Record observations for all of the steps (I-V) of the copper cycle in your lab book. Be sure to label each step (I-V). The observations for each step should include:

- the appearance of the reactants before the reaction
- the appearance of the reactants ***during*** the reaction (for example, bubbles, flames, etc.)
- the appearance of the products after the reaction.

Your observations should include state(s) of matter, color, texture, smell, etc. where applicable. If your observations are not detailed, you may not receive full credit.

One step also requires a specific chemical test using litmus paper to check for acidity. Be sure to also record the results of these tests in your lab notebook.

**You will turn in worksheet pages 6-7 along with the copies of your observations from your lab notebook.

## *Step I: Procedure* - Oxidizing Cu with concentrated nitric acid, $HNO_3$*(aq)*

1. Place a sample of weighing paper in the balance. Tare the balance, so it reads 0.0000 g. Use forceps to transfer about 0.35-0.40 g of Cu strips onto the weighing paper. Record the mass of the Cu strips. Transfer the Cu strips into a clean 250-mL beaker labeled with one of your group member's initials. Record the appearance of the copper metal in your lab report.

**CAUTION: Concentrated nitric acid is highly corrosive, so it can cause severe chemical burns and damage clothing. Handle with care and avoid breathing the fumes. Any nitric acid spilled**

**on skin must be rinsed immediately with water for 15 minutes. Any acid spilled on your work area must be neutralized then the entire area should be washed and dried.**

**CAUTION: Concentrated nitric acid reacts with copper metal to form brown *toxic* $NO_2$ gas. Leave the reaction beaker in the fume hood until all of the brown gas is vented in the hood.**

2. **In a fume hood**, use a 10-mL graduated cylinder to carefully measure about 3 mL of concentrated nitric acid, $HNO_3(aq)$. Slowly pour the nitric acid onto the Cu strips in the beaker, swirling the beaker to maximize contact between the Cu and nitric acid until all of the solid Cu has dissolved and the $NO_2$ gas has escaped. ***Keep the reaction beaker in the hood until all the toxic brown $NO_2$ gas is gone, and keep your face away from the hood to avoid inhaling nitric acid fumes and $NO_2$ gas.*** Describe the reaction between $HNO_3$ and the Cu metal in your lab report.

3. Dilute the resulting solution with about 10 mL of deionized water. Describe the appearance of the resulting solution containing $Cu^{2+}$ in your data table.

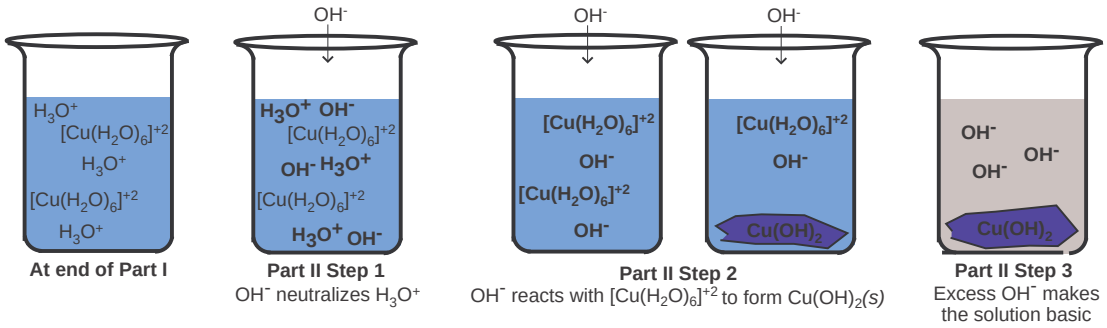
## *Step II: Chemistry* - Precipitating $Cu(OH)_2(s)$ with $NaOH(aq)$

In Part II, two reactions are carried out by adding $NaOH(aq)$. In the first reaction, the hydroxide ions ($OH^-$) from the $NaOH(aq)$ neutralize the excess hydronium ions ($H_3O^+$) left over from the previous part.

Once all the $H_3O^+$ ions are neutralized, additional $OH^-$ ions react with the $Cu^{2+}$ complex ion to form a gelatinous blue $Cu(OH)_2$ precipitate.

Once all the $Cu^{2+}$ ions have reacted, no more precipitate forms. Adding more $OH^-$ ions makes the solution basic, so it can turn red litmus paper blue. The picture sequence on the next page outlines the step-by-step process that occurs during this step.

**Figure 2: Step-wise Illustration of the Precipitation of $Cu(OH)_2$ in Part II**




**1st Beaker**: At the end of Part I, hydrated copper complex, $Cu^{2+}$ are present, making the solution blue, and excess hydronium ions ($H_3O^+$) remain from the nitric acid used.

**2nd Beaker**: Adding $NaOH(aq)$ to the blue solution results in the $OH^-$ ions neutralizing the $H_3O^+$ ions to form water: $H_3O^+(aq) + OH^-(aq) \rightarrow 2\ H_2O(l)$. The $Na^+$ ions and resulting water molecules are not shown.

**3rd and 4th Beakers**: Once all the $H_3O^+$ are neutralized, adding more $NaOH(aq)$ results in the $OH^-$ ions reacting with the $Cu^{2+}$ to form the blue $Cu(OH)_2(s)$ precipitate shown at the bottom of the beaker. Water molecules released from the complex ion are not shown.

**5th Beaker**: When all of the $Cu^{2+}$ ions have been converted to $Cu(OH)_2(s)$ precipitate, adding more $NaOH(aq)$ results in unreacted $OH^-$ ions in solution, which makes the solution basic. Red

litmus paper can be used to confirm the solution is basic. Note that the solution is no longer blue since no $Cu^{2+}$ ions are present in the solution.

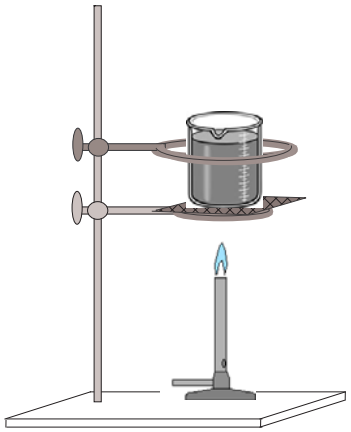
## *Step II: Procedure* - Precipitating $Cu(OH)_2$ with NaOH solution

**CAUTION: Sodium hydroxide (NaOH) can easily damage eyes. It is corrosive and can cause chemical burns and damage clothing. Any NaOH splashed into eyes or spilled on skin must be rinsed immediately with water for 15 minutes. Any base spilled on your work area must be neutralized then the entire area should be washed and dried.**

1. While constantly stirring the Cu solution, slowly add 6M NaOH*(aq)* from the dropper bottles. First, the $OH^-$ from the NaOH added will neutralize the excess acid left over from Part I.
2. Once all the acid is neutralized, additional $OH^-$ ions react with the $Cu^{2+}$ to form $Cu(OH)_2$*(s)*, a blue precipitate. Record what you observe in your lab report.

When adding more NaOH does not produce more precipitate, the solution can be tested to determine if the $Cu^{2+}$ has been precipitated and additional $OH^-$ has made the solution basic. Use red litmus paper to test if the solution is basic as follows. *Without disturbing any precipitate*, use a glass stir rod to place a drop of **solution** (NOT the precipitate) on a piece of red litmus paper. If it turns blue, the solution is bas Stop adding NaOH when the solution turns red litmus paper blue. Describe your litmus test in your lab report.

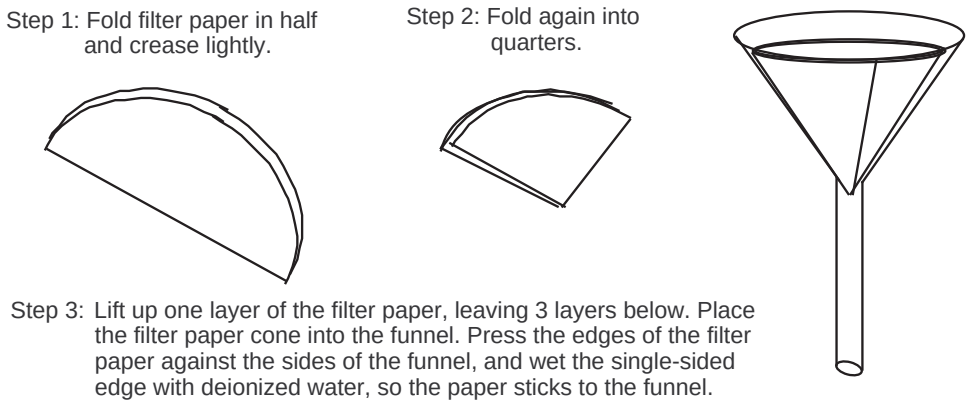
## *Step III: Procedure* - Converting $Cu(OH)_2$*(s)* to CuO*(s)*



1. Set up a ring stand as shown in the figure at the right. Set up a ring clamp, and put a wire gauze on top of it. Above it, attach another ring clamp with a diameter large enough to go around a 250-mL beaker. You are going to set your 250 mL beaker on the lower ring and gauze. The upper clamp will hold the beaker in place so it does not fall.

2. Add about 80-90 mL of deionized water to your reaction beaker from Part II. Carefully place the beaker on the ring stand inside the upper ring. **CAUTION:** Gently heat the beaker over a **low** flame. (Set the inner cone of the Bunsen burner flame to a height of about 1 inch and the lower ring stand about 4 inches above that (5-6 inches above the top o the Bunsen burner)). Constantly stir the solution with the glass end of the stirring rod until all the blue precipitate turns black, and the solution is clear. If the solution starts to bump or boil, immediately remove the beaker from the heat and let the solution cool slightly. Describe what happens to the $Cu(OH)_2$ precipitate upon heating in your lab report.

3. Allow the beaker and contents to cool. While they are cooling, set up the gravity filtration apparatus Obtain a second ring stand, and attach a ring clamp that is small enough to hold the plastic funnel. Prepare the filter paper as shown below:




Finally, place the plastic funnel in the small ring clamp, and place a 400-mL beaker beneath it to collect the filtrate (the liquid that goes through the filter paper). The funnel's stem should be just inside the beaker to prevent splashing.

4. Use the markings on a clean 150-mL beaker to measure out about 25 mL of deionized water. Boil the water on a hotplate to wash the precipitate in step 6.
5. When the 250-mL reaction beaker has cooled to room temperature, pour the CuO precipitate into the funnel to filter the contents. Transfer the last traces of the solid from the reaction beaker into the funnel, using a stream of deionized water.
6. Use a disposable pipet to wash the precipitate on the filter paper using the hot deionized water heated in the 150-mL beaker. Allow each portion of hot water to drain through the filter paper into the beaker below before adding the next portion. Use all 25 mL of the hot deionized water to thoroughly wash the CuO precipitate.
7. Wash the 250-mL beaker, and rinse with deionized water. Replace the 400-mL beaker under the filter funnel with the clean 250-mL beaker. Discard the filtrate (solution) collected in the 400-mL beaker into the properly labeled waste container. Clean and dry the 400 mL beaker for use in Part V. ***Keep the CuO solid in the filter paper for use in Part IV.***

## *Step IV: Procedure* - Dissolving CuO(s) with sulfuric acid, $H_2SO_4$*(aq)*

**CAUTION: Sulfuric acid, $H_2SO_4$*(aq)*, is corrosive, so it can cause severe chemical burns and damage clothing. Handle with care and avoid breathing the fumes. Any sulfuric acid spilled on skin must be rinsed immediately with water for 15 minutes. Any acid spilled on your work area must be neutralized, and the entire area should be washed and dried.**

1. Add about 10 mL of 3M sulfuric acid, $H_2SO_4$ (check the label before pouring), to the funnel to dissolve the CuO precipitate. Allow the solution to drain through the funnel to the rinsed 250-mL beaker. Repeat the procedure until all of the CuO solid dissolves to $Cu^{2+}$ ions. Use as little of the sulfuric acid as possible in this step. Describe the reaction between the CuO precipitate and the $H_2SO_4$ in your lab report.
2. Use your water bottle to wash the last traces of solution from the empty funnel into the 250-mL beaker which now contains the acid solution and aqueous $Cu^{2+}$ ions. **Keep this resulting solution for use in the Part V.**

## *Step V: Procedure* - Reducing $Cu^{2+}$ ions with Zn Metal

1. Use a weighing boat to measure and transfer about 1 g of Zn mesh to the $Cu^{2+}$ solution in the 250-mL beaker. Constantly stir the mixture with a ***glass stirring rod***. **Do not use any metal object that will react with the acid to stir the solution.** Continue stirring until all the $Cu^{2+}$ ions have been reduced to Cu metal as indicated by the solution becoming colorless. Dissolve any excess Zn by adding a few drops of 3M $H_2SO_4$*(aq)*. Describe your observations of the reduction of $Cu^{2+}$ to Cu metal.
2. Allow the Cu metal to settle at the bottom of the beaker. Without losing any of the solid, carefully decant (pour off) as much of the supernatant liquid as possible into a 400-mL beaker. Some liquid will remain in the first beaker with the Cu metal. Wash the Cu metal 3 times using 20-mL portions of deionized water by stirring and then allowing the solid to re-settle. Again, decant the liquid into the 400-mL beaker each time.
3. Weigh a clean, dry evaporating dish. Transfer the Cu metal and any remaining water into the evaporating dish using a stream of deionized water. Decant most of the water from the evaporating dish. Use a disposable pipet to remove as much remaining water from the evaporating dish without losing any solid.
4. Write one of your group members' names on a folded piece of paper towel. Place your group's evaporating dish on the paper towel in the oven (between the hoods) to let the Cu completely dry. Check it after about 10 minutes. If the copper pieces are loose then it is dry. If it appears black in color, then the copper has been heated too much and has turned to copper (II) oxide.
5. When the Cu appears completely dry, let the evaporating dish cool to room temperature, and weigh the evaporating dish with the Cu. Record the final mass in your lab report.

**Wash and dry all of your glassware, equipment, and your lab area to prevent chemical contamination and potential hazards.**

## *Calculating Percent Copper Recovered*

Theoretically, the mass of Cu recovered should be equal to the mass of the original Cu sample. The overall efficiency of the experiment is measured by calculating the percentage of copper recovered:

$$\textbf{percent recovered} = \frac{\textbf{mass of final product}}{\textbf{mass of initial sample}} \times \textbf{100\%} \qquad [11]$$

Ideally, the percent recovered should be close to 100%, which indicates that most (if not all) of the copper was successfully transformed through all five parts of the experiment.

# *The Copper Cycle*

Name: _____

Partner: _____

Section Number: _____

*****Turn in pages 6-7 along with your lab notebook copies*****

**For each equation shown below (1 for each step of the copper cycle), indicate**

A. the products of the equation, include phases of all products,
B. balance the equation, and
C. tell what type of equation it is: synthesis, decomposition, single replacement, or double replacement (none of them is combustion)

**Step I.** ___ $Cu_{(s)}$ + ____$HNO_{3\,(aq)}$ → ____$NO_{2\,(g)}$ + ____$H_2O_{(l)}$ + _____

Think about what the last product should be…it is logical, unlike the $NO_2$ or $H_2O$.

type of reaction: _____

**Step II.** ___$Cu(NO_3)_{2\,(aq)}$ + ____$NaOH_{(aq)}$ →

type of reaction: _____

**Step III.** ____$Cu(OH)_{2\,(s)}$ + heat → ____$CuO_{(s)}$ + ____$H_2O_{(l)}$
(This is actually a weird step that does not have a nice easy equation.)

type of reaction: _____

**Step IV.** ____$CuO_{(s)}$ +____ $H_2SO_{4\,(aq)}$ → _____ + _____

type of reaction: _____

**Step V.** ____$CuSO_{4\,(aq)}$ + ____$Zn_{(s)}$ → _____ + _____

type of reaction: _____

**POST-LAB QUESTIONS:**

1) Percent Copper Recovered

A student performing this experiment started with a 0.3769 g sample of copper turnings, which was dissolved in concentrated nitric acid. After completing the series of reactions, the student isolated 0.3492 g of copper. Calculate the percent copper recovered by the student.

2) Indicate whether the following procedural errors would result in an **incorrectly high** or **incorrectly low** percent recovery. **Circle and explain your answer.**

a. The solution was not basic before being heated in Part III.
High Low

b. In Part III, the solution was poured into the funnel until it went above the top of the filter paper, and some black solid was disposed of with the filtrate.
High Low

c. The solution decanted in Part V was slightly blue in color.
High Low

d. After all the copper metal was obtained in Part V, it took too long for the excess Zn granules to dissolve, so a student added concentrated nitric acid to the solution, resulting in a brown gas.
High Low

e. In Part V, the copper metal was weighed after it turned dark brown/red.
High Low