# Department of Chemistry Outreach UNB Fredericton

### **Copper Cycle**

This experiment is based on the experiment "Chemistry of Copper" in J.A. Beran's *Laboratory Manual for Principles of General Chemistry*, 2009, John Wiley & Sons, Inc.

### **INTRODUCTION**

Copper is one of the most used metals due to its relative chemical inertness (does not react with air, moisture, dilute acids and bases). Canada is a major producer of copper, used in the manufacture of coins and electric wirings to name a few uses. This experiment will explore some of the very diverse chemistry of copper and its compounds through a variety of redox, precipitation, decomposition and acid-base reactions. You will also learn that some compounds cannot be obtained in a single step such as compound L below:

 $A+B \rightarrow L$ 

But may require many steps as indicated below:

 $A + B \rightarrow C$  $C + D \rightarrow E$  $E + F \rightarrow G$  $G + H \rightarrow I$  $I + K \rightarrow L$ 

This can also be written as follows:

$$A \xrightarrow{B} C \xrightarrow{D} E \xrightarrow{F} G \xrightarrow{H} I \xrightarrow{K} L$$

This would be a five step reaction. Sometimes you have to isolate and purify the products in each step, sometimes you can just keep on going. We will explore a five step reaction which is called the copper cycle because you start and finish with the same material, metallic copper.

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$$Cu \xrightarrow{HNO_3} Cu(NO_3)_2 \xrightarrow{NaOH} Cu(OH)_2 \xrightarrow{\Delta} CuO \xrightarrow{H_2SO_4} CuSO_4 \xrightarrow{Mg} CUSO_4 \xrightarrow$$

#### Scheme 1

One thing one has to be very careful with is that chemists usually write balanced equations. That would be very difficult to do in a multi step reaction as in Scheme 1 (that's why we do not label it as Equation 1). We can however break down the five steps as follows and use balanced equations:

$\operatorname{Cu}(s) + 4 \operatorname{HNO}_3(aq) \to \operatorname{Cu}(\operatorname{NO}_3)_2(aq) + 2 \operatorname{NO}_2(g) + 2 \operatorname{H}_2\operatorname{O}(l)$	(Eq 1)
$\operatorname{Cu}(\operatorname{NO}_3)_2(aq) + 2 \operatorname{NaOH}(aq) \rightarrow \operatorname{Cu}(\operatorname{OH})_2(s) + 2 \operatorname{NaNO}_3(aq)$	(Eq 2)
$\operatorname{Cu}(\operatorname{OH})_2(s) \xrightarrow{\Delta} \operatorname{CuO}(s) + \operatorname{H}_2\operatorname{O}(g)$	(Eq 3)
$CuO(s) + H_2SO_4(aq) \rightarrow CuSO_4(aq) + H_2O(l)$	(Eq 4)
$CuSO_4(aq) + Mg(s) \rightarrow Cu(s) + MgSO_4(aq)$	(Eq 5)

#### PROCEDURE

#### **Step 1: Preparation of Cu(NO<sub>3</sub>)2:**

- 1. Add 10 Cu pellets into a clean and dry centrifuge tube. Use a test tube clamp to hold the tube using a test tube clamp for the remainder of the experiment.
- 2. IN A FUME HOOD, add 10 drops of conc HNO<sub>3</sub> drop wise and very slowly until no further evidence of a chemical reaction is observed. Do not add an excess of HNO<sub>3</sub> or you will have trouble in the next step when you neutralize and react with NaOH! The gas that evolves is NO<sub>2</sub> which is highly toxic. Keep the fume hood sash as low as possible. Record the colour of the gas and the solution at this point.

# Step 2: Preparation of Cu(OH)2:

3. Take the centrifuge tube to your bench (unless your group works in a fumehood) and place in front of an exhaust arm. Using a stir rod, stir the solution while adding 10

drops of 6 M NaOH. A precipitate will be observed. After a couple of minutes add 10 more drops of NaOH solution and rinse the stirring rod with water from a wash bottle making sure that all washes enter the centrifuge tube.

4. Centrifuge the mixture for 1 minute (ask your TA for operating instructions). Add 3 more drops of NaOH solution to the supernatant. If additional precipitate forms you probably added too much HNO<sub>3</sub> in the previous step. In that case add 7 more drops of NaOH solution and repeat the centrifugation. Test again by adding 3 more drops of NaOH solution. Repeat this test and centrifugation until no further precipitate is observed. At this point record the colour of the precipitate and supernatant.

#### **Step 3: Preparation of CuO:**

5. Decant the supernatant into a small beaker and take the tube to the fume hood with the sand bath. Holding the tube with its opening pointing towards the back of the fume hood (**never point at yourself or anyone else**), slowly lower the tube into the sand first by resting it on the surface. Heat until the solid changes colour then immerse the tube into the sand. Make sure none of the solid is ejected due to rapid heating, otherwise you will have to start from step 1! The solid does not need to be heated to dryness to completely convert to the oxide. At this point record the colour of the solid.

#### **Step 4: Preparation of CuSO4:**

6. Take your centrifuge tube with the CuO back to your bench (unless your group works in a fumehood) and place in front of an exhaust arm and add 20 drops of 6 M H<sub>2</sub>SO<sub>4</sub>. The residue will react to give a soluble product. At this point record the colour of the solution. If your solid does not react well, take it to the fume hood with the sand bath and gently heat it by immersing the tube in the sand (**Be careful of the hot sand**).

#### **Step 5: Preparation of Cu:**

 Dilute the CuSO<sub>4</sub> solution with RO water until the test tube is half filled. Add a 1 cm piece of Mg ribbon and observe the changes until all of the Mg has reacted. Add more Mg ribbon until the solution is no longer blue. At this point record the colour of the coating you saw on the Mg ribbon. Do you see the evolution of a gas? If a milky white precipitate of  $Mg(OH)_2$  is observed, add a few drops of  $H_2SO_4$  and break up the coating on the Mg ribbon with a stir rod. Add 10 drops of 6 M  $H_2SO_4$  to remove the excess Mg by converting it into soluble MgSO<sub>4</sub>. Wait until the evolution of gas has subsided.

- 8. Centrifuge for 1 minute, decant and discard the supernatant into a separate beaker that you've labeled as your waste beaker. Add approximately 1 mL of water to the solid, stir the mixture with a rod, centrifuge and decant the supernatant into your waste beaker. Repeat the washing 2 more times.
- 9. Record the colour of the solid.

# REPORT

#### Names: \_\_\_\_\_

# Table 1. Experimental Results and Observations

Step 1		
Number of Cu pellets		
Appearance of Cu pellets		
Colour of the gas		
Colour of the solution		
Step 2		
# of drops of NaOH solution that were added in total		
Colour of the precipitate		
Colour of the supernatant		
Step 3		
Is a gas given off during heating?		
What is the gas?		
Colour of the solid		
Step 4		
Colour of the solution		
Step 5		
Colour of the coating on the Mg ribbon		
Is a gas given off during the reaction?		
What is the gas?		
Appearance of the final product		