CHEMICAL REACTIONS OF COPPER AND PERCENT YIELD KEY

Objective

To gain familiarity with basic laboratory procedures, some chemistry of a typical transition element, and the concept of percent yield.

Apparatus and Chemicals

0.5 g piece of no. 16 or no. 18 copper wire	evaporating dish
250 mL beaker (2)	weighing paper
concentrated HNO_3 (4 – 6 mL)	6.0 M H_2SO_4 (15 mL)
graduated cylinder	granular zinc
3.0 M NaOH (30 mL)	methanol
carborundum boiling chips	acetone
stirring rod	towel
iron ring and ring stand	balance
wire gauze	aluminum foil cut in 1-inch squares
Bunsen burner	concentrated HCl (drops)

Discussion

Most chemical synthesis involves separation and purification of the desired product from unwanted side products. Some methods of separation, such as filtration, sedimentation, decantation, extraction, and sublimation were discussed earlier. This experiment is designed as a quantitative evaluation of your individual laboratory skills in carrying out some of these operations. At the same time you will become more acquainted with two fundamental types of chemical reactions - redox reactions and metathesis (double-displacement) reactions. By means of these reactions, you will finally recover the copper sample with *maximum efficiency*. The chemical reactions involved are the following.

$Cu(s) + 4\,HNO_3(aq) \longrightarrow Cu(NO_3)_2(aq) + 2\,NO_2(g) + 2\,H_2O(l)$	Redox	[1]
$Cu(NO_3)_2(aq) + 2\,NaOH(aq) \longrightarrow Cu(OH)_2(s) + 2\,NaNO_3(aq)$	Metathesis	[2]
$Cu(OH)_2(s) \longrightarrow CuO(s) + H_2O(g)$	Dehydration	[3]
$CuO(s) + H_2SO_4(aq) \longrightarrow CuSO_4(aq) + H_2O(l)$	Metathesis	[4]
$CuSO_4(aq) + Zn(s) \longrightarrow ZnSO_4(aq) + Cu(s)$	Redox	[5]

Each of these reactions proceeds to completion. Metathesis reactions proceed to completion whenever one of the components is removed from the solution, such as in the formation of a gas or an insoluble precipitate (driving forces). This is the case for reaction [1], [2], and [3], where in reactions [1] and [3] a gas and in reaction [2] an insoluble precipitate are formed. Reaction [5] proceeds to completion because zinc has a lower ionization energy or oxidation potential that copper.

The objective in this experiment is to recover all of the copper you begin with in analytically pure form. This is the test of your laboratory skills.

The percent yield of the copper can be expressed as the ratio of the recovered weight to initial weight, multiplied by 100:

$$\% \text{ yield} = \frac{\text{recovered weight of Cu}}{\text{initial weight of Cu}} \times 100$$

Procedure

- Weight approximately 0.500 g of no. 16 or no. 18 copper wire (1) to the nearest 0.0001 g and place it in a 250 mL beaker. Add 4-5 mL of concentrated HNO_3 to the beaker, IN THE HOOD. After the reaction is complete, add 100 mL distilled H_2O. Describe the reaction (6) as to color change, evolution of gas, and change in temperature (exothermic or endothermic) in the report sheet.
- Add 30 mL of 3.0 *M* NaOH to the solution in your beaker and describe the reaction (7). Add two or three boiling chips and carefully heat the solution -- while stirring with a glass stirring rod -- just to the boiling point. Describe the reaction on your report sheet (8). *Remove the boiling chips.*
- Allow the black CuO to settle; then decant the supernantant liquid. Add about 200 mL of very hot distilled water and allow the CuO to settle. Decant once more. What are you removing by washing and decanting (9)?
- Add 15 mL of 6.0 *M* H_2SO_4. What copper compound is present in the beaker now (10)?

Your instructor will tell you whether you should use Zn or Al for the reduction of Cu (II) in the following step.

A. Zinc
In the hood, add 2.0 g of 30-mesh zinc metal all at once and stir until the supernatant liquid is colorless. Describe the reaction on your report sheet (11). What is present in solution (12)? When gas evolution has become *very* slow, heat the solution gently (but do not boil) and allow it to cool. What gas is formed in this reaction (13)? How do you know (14)?

B. **Aluminum**
In the hood, add several 1-inch squares of aluminum foil and a few drops of concentrated HCl. Continue to add pieces of aluminum until the supernatant liquid is colorless. Describe the reaction on your report sheet (11). What is present in solution (12)? What gas is formed in this reaction (13)? How do you know (14)?

When gas evolution has ceased, decant the solution and transfer the precipitate to a preweighed porcelain evaporating dish (3). Wash the precipitated copper with about 5 mL of distilled water, allow to settle, decant the solution, and repeat the process. What are you removing by washing (15)? Wash the precipitate with about 5 mL of methanol (KEEP THE METHANOL AWAY FROM FLAMES _ IT IS FLAMMABLE!) Allow the precipitate to settle, and decant the methanol. (METHANOL IS ALSO EXTREMELY TOXIC: AVOID BREATHING THE VAPORS AS MUCH AS POSSIBLE.) Finally, wash the precipitate with about 5 mL of acetone (KEEP THE ACETONE AWAY FROM FLAMES - IT IS EXTREMELY FLAMMABLE!), allow the precipitate to settle, and decant the acetone from the precipitate. Prepare a steam bath as illustrated and dry the product on your steam bath for at least 5 minutes.

Wipe the bottom of the evaporating dish with a towel, remove the boiling chips and weigh the evaporating dish plus copper (2). Calculate the final a\weight of copper (4). Compare the weight with your initial weight and calculate the percent yield (5). What color is your copper sample (16)? Is it uniform in appearance (17)? Suggest possible sources of error in this experiment (18).

Chemical Reactions of Copper and Percent Yield KEY

Pre-lab (Review Questions)

1. Give an example, other than the ones listed in this experiment, of redox and metathesis reactions.

Redox: $Zn + MgCl_2 \longrightarrow ZnCl_2 + Mg$ Zinc is oxidized: $Zn^0 \longrightarrow Zn^{2+} + 2e^-$

Magnesium is reduced: $Mg^{2+} + 2e^- \longrightarrow Mg^0$

Metathesis: $Pb(NO_3)_2 + H_2S \longrightarrow PbS + HNO_3$ (metathesis is also known as a double-replacement reaction)

2. When will reactions proceed to completion?

Driving forces for double replacement reaction is formation of water, gas or a solid.

Single replacement reactions we use an activity series to predict if they will occur.

For a reaction to proceed to completion all of the reactants must mix: they may need to be stirred, or heated to assist in the process of them reacting.

3. Define percent yield in general terms.

Percent yield is a measure of how well the reaction proceeded to completion. The formula for percent yield is the experimental yield divided by the calculated (theoretical yield).

4. Name six methods of separating materials.

a) filtration b) magnetism c) centrifugation d) decantation e) color f) distillation

5. Give criteria in terms of temperature changes for exothermic and endothermic reactions.

Exothermic reactions - release heat and feel "hot" to the touch

Endothermic reaction - gain heat and feel "cold" to the touch

6. If 1.65 g of $Cu(NO_3)_2$ are obtained from allowing 0.93 g of Cu to react with excess HNO_3, what is the percent yield of the reaction?

$$\% = \frac{1.65\text{ g}}{2.75\text{ g}}$$

1.65 g

$$\mathbf{Cu + 2\,HNO_3 \longrightarrow Cu(NO_3)_2 + H_2}$$

0.93 g excess x g

$\% = 60\%$ yield

$$x\text{ g } Cu(NO_3)_2 = 0.93\text{ g Cu}\left(\frac{1\text{ mol Cu}}{63.5\text{ g Cu}}\right)\left(\frac{1\text{ mol } Cu(NO_3)_2}{1\text{ mol Cu}}\right)\left(\frac{187.5\text{ g } Cu(NO_3)_2}{1\text{ mol } Cu(NO_3)_2}\right) = 2.75\text{ g } Cu(NO_3)_2$$

7. What is the maximum percent yield in any reaction?

100%; any value higher would be impurities in product (e.g. water, by-product)

8. What is meant by the terms *decantation* and *filtration*?

Decantation - pour off solvent leaving behind precipitate

Filtration - pass through filter that separates our components of a mixture by differences in particle size

9. When $Cu(OH)_2(s)$ is heated, Copper (II) oxide and water are formed.
   Write a balanced equation for the reaction.

$$Cu(OH)_2(s) \longrightarrow CuO(s) + H_2O(g)$$

10. When sulfuric acid and copper (II) oxide are allowed to react, copper (II) sulfate and water are formed.
    Write a balanced equation for this reaction.

$$H_2SO_4(aq) + CuO(s) \longrightarrow CuSO_4(aq) + H_2O(l)$$

11. When copper (II) sulfate and aluminum are allowed to react, aluminum sulfate and copper are formed.
    What kind of reaction is this? Write a balanced equation for this reaction.

$$3\ CuSO_4(aq) + 2\ Al(s) \longrightarrow Al_2(SO_4)_3(aq) + 3\ Cu(s)$$

This reaction is an example of a redox reaction: where aluminum is oxidized and copper is reduced. The copper is the oxidizing agent and the aluminum is the reducing agent.

Aluminum is oxidized: $Al^0 \longrightarrow Al^{3+} + 3e^-$ Copper is reduced: $Cu^{2+} + 2e^- \longrightarrow Cu^0$

It could also be called a single replacement reaction – where aluminum is chemically more active than copper.

REPORT SHEET KEY

Chemical Reactions of Copper and Percent Yield

1. Weight copper initial ______________
2. Weight of copper and evaporating dish ______________
3. Weight of evaporating dish ______________
4. Weight of copper final ______________
5. % Yield (show calculations) ______________

6. Describe the reaction $Cu(s) + HNO_3(aq) \longrightarrow$ **$Cu(NO_3)_2(aq) + 2\ NO_2(g) + 2\ H_2O(l)$**

   The addition of nitric acid caused the copper metal to slowly dissolve. A red-brown gas (NO_2) was produced. The odor was similar to the smell of chlorine. The nitric acid liquid changed color from colorless to a blue-green color.

   $Cu(s) + 4\ HNO_3(aq) \longrightarrow Cu(NO_3)_2(aq) + 2\ NO_2(g) + 2\ H_2O(l)$

7. Describe the reaction $Cu(NO_3)_2(aq) + NaOH(aq) \longrightarrow$ **$Cu(OH)_2(s) + 2\ NaNO_3(aq)$**

   The addition of sodium hydroxide solution with the copper nitrate solution produced a bright blue colored solid (gel-like) material [$Cu(OH)_2$].

   $Cu(NO_3)_2(aq) + 2\ NaOH(aq) \longrightarrow Cu(OH)_2(s) + 2\ NaNO_3(aq)$

8. Describe the reaction $Cu(OH)_2(s) \longrightarrow$ **$CuO(s) + H_2O(g)$**

   When the beaker containing the copper (II) hydroxide and water was heated the blue solid changed color into a black fine powder. Upon cooling the black powder (CuO) could be separated from the water by decantation.

   $Cu(OH)_2(s) \longrightarrow CuO(s) + H_2O(g)$

9. What are you removing by this washing?

   Unreacted (impurities) and excess $NaNO_3$ that wasn't removed in the previous step.

10. What copper compound is present in the beaker?

    $CuSO_4$ copper (II) sulfate

11. Describe the reaction $CuSO_4(aq) + Zn(s)$, or $CuSO_4(aq) + Al(s)$

    When aluminum foil is added to the solution of copper (II) sulfate, the foil dissolves and has copper spots. The solution changes color from pale blue to colorless. A gas is released (hydrogen).

12. What is present in solution?

    Al^{3+} ions and SO_4^{2-} ions. They are removed in the final washing to leave you with pure Copper!

Chemical Reactions of Copper and Percent Yield

13. What is the gas?

    Hydrogen gas.

14. How do you know?

    Any time an acid is added to a metal hydrogen gas is released. We also checked with a glowing wooden splint that burst into flames when placed in the beaker.

15. What are you removing by washing?

    Al^{3+} ions and SO_4^{2-} ions.

16. What color is your copper sample?

    Initial color is red-brown (copper colored) when wet. After drying, many samples will have a white residue of aluminum sulfate crystals that weren't removed during washing. Small bits of excess aluminum may be present giving the silver color of aluminum mixed in with copper sample. Finally, upon standing, the copper may oxidize and change to a slightly green color.

17. Is it uniform in appearance?

    Yes, with exceptions listed in question #16 (above).

18. Suggest possible sources of error in this experiment.

    When decanting, lost some sample by pouring it out. It is difficult to have all CuO settle to bottom and remove all liquid.

    Approximate volumes of acids and bases were added. We assumed we had excess in all cases and removed the excess by washing.

    Extremely hard to remove excess Al foil – addition of [hydrochloric acid] to dissolve foil may have caused the copper product to react and form copper (II) chloride.

POST LAB QUESTIONS KEY

1. If your percent yield of copper was greater than 100%, what are two plausible errors you may have made?

   Sample was not fully dried and still contained water. Impurities were mixed in with copper causing it to weigh more than it should.

2. Consider the combustion of methane, CH_4:

$$CH_4(g) + 2\,O_2(g) \;\text{----->}\; CO_2(g) + 2\,H_2O(g)$$

Suppose 2 mole of methane is allowed to react with 3 mol of oxygen.

a) What is the limiting reagent? (show work)

   OXYGEN is the limiting reactant. According to the balanced chemical equation, you need 2x the amount of oxygen as methane. You would need 4 mole of oxygen to react with 2 mol of methane (you have only 3 mol of oxygen).

b) How many moles of CO_2 can be made from this mixture? How many grams of CO_2?

$$x \text{ mol } CO_2 = 3 \text{ mol } O_2 \left(\frac{1 \text{ mol } CO_2}{2 \text{ mol } O_2}\right) = 1.5 \text{ mol } CO_2$$

$$x \text{ g } CO_2 = 3 \text{ mol } O_2 \left(\frac{1 \text{ mol } CO_2}{2 \text{ mol } O_2}\right)\left(\frac{44 \text{ g } CO_2}{1 \text{ mol } CO_2}\right) = 66 \text{ g } CO_2$$

3. Suppose 8.00 g of CH_4 is allowed to burn in the presence of 6.00 g of oxygen.
   How much (in grams) CH_4, O_2, CO_2, and H_2O remain after the reaction is complete?

limiting

$$CH_4(g) + 2\,O_2(g) \;\text{----->}\; CO_2(g) + 2\,H_2O(g)$$

CH_4	O_2
8 g	6 g
$\downarrow$ / 16 g	$\downarrow$ / 32 g
$\dfrac{0.5 \text{ mol } CH_4}{1}$	$\dfrac{\mathbf{0.1875 \text{ mol } O_2}}{2}$
0.5	0.09375

[Have **0 g O_2** remaining]

$$x \text{ g } CH_4 = 0.1875 \text{ mol } O_2 \left(\frac{1 \text{ mol } CH_4}{2 \text{ mol } O_2}\right)\left(\frac{16 \text{ g } CH_4}{1 \text{ mol } CH_4}\right) = 1.5 \text{ g } CH_4$$

[Have **6.5 g CH_4** remaining]

$$x \text{ g } CO_2 = 0.1875 \text{ mol } O_2 \left(\frac{1 \text{ mol } CO_2}{2 \text{ mol } O_2}\right)\left(\frac{44 \text{ g } CO_2}{1 \text{ mol } CO_2}\right) = 4.125 \text{ g } CO_2$$

[Have **4.125 g CO_2** produced]

$$x \text{ g } H_2O = 0.1875 \text{ mol } O_2 \left(\frac{2 \text{ mol } H_2O}{2 \text{ mol } O_2}\right)\left(\frac{18 \text{ g } H_2O}{1 \text{ mol } H_2O}\right) = 3.375 \text{ g } H_2O$$

[Have **3.375 g H_2O** produced]

4. How many milliliters of 6.0 *M* H_2SO_4 are required to react with 0.80 g of CuO according to Equation [4]?

$$\mathbf{CuO(s)} + \mathbf{H_2SO_4(aq)} \longrightarrow \mathbf{CuSO_4(aq)} + \mathbf{H_2O(l)}$$

0.80 g (CuO)   x mol (H_2SO_4)

$$x \text{ mol } H_2SO_4 = 0.80 \text{ g CuO} \left(\frac{1 \text{ mol CuO}}{79.5 \text{ g CuO}}\right)\left(\frac{1 \text{ mol } H_2SO_4}{1 \text{ mol CuO}}\right) = 0.0100628 \text{ mol } H_2SO_4$$

convert to millimoles (mmol) by dividing by 1000.

$$\text{Molarity (M)} = \frac{\text{mol solute}}{\text{L of solution}} \Rightarrow 6.0 \text{ M } H_2SO_4 = \frac{10.0628 \text{ mmol}}{\text{X mL of solution}} = \mathbf{1.677 \text{ mL } 6.0 \text{ M } H_2SO_4}$$

5. If 2.00 g of Zn is allowed to react with 1.75 g of $CuSO_4$ according to Equation [5], how many grams of Zn will remain after the reaction is complete? *(from the question we can assume that Zn in excess reactant)*

$$\mathbf{CuSO_4(aq)} + \overset{\text{excess}}{\mathbf{Zn(s)}} \longrightarrow \mathbf{ZnSO_4(aq)} + \mathbf{Cu(s)}$$

1.75 g (CuSO$_4$)   2.00 g (Zn)

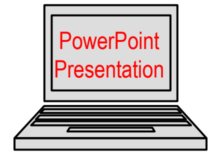
x g

$1.75 \text{ g} \xrightarrow{/\ 159.5 \text{ g}} 0.01097 \text{ mol} \longrightarrow 0.01097 \text{ mol} \xrightarrow{\times\ 65.4 \text{ g/mol Zn}} x = 0.72 \text{ g}$

The reaction will consume 0.72 g of Zn. Therefore you will have 2.00 g – 0.72 g or **1.28 g of Zn remaining**.

6. What is meant by the term limiting reagent?

The limiting reactant is the starting material used in a chemical reaction that is used up first (or that you run out of). When no more of it remains, no additional product can be made and the reaction stops. The quantity of limiting reactant determines (LIMITS) the amount of product that is made.



TEACHER NOTES:

Warning: This lab experiment requires a large quantity or reagents. The acids and base are very *concentrated* and should only be used in a fume hood with proper teacher supervision.

For a class of 100 students you will need:

750 mL concentrated nitric acid [HNO_3]
650 mL concentrated sulfuric acid [H_2SO_4]
480 g sodium hydroxide (NaOH)

The concentrated nitric acid is not diluted.

The concentrated sulfuric acid is diluted to 6 M H_2SO_4.
Concentrated sulfuric acid is 18.1 M. Therefore, take 1 part [H_2SO_4] to 2 part H_2O.
The resulting solution will be approximately 6 M H_2SO_4.

To make 3 M NaOH, begin with 2000 mL of cold distilled water and add 240 g NaOH.
You will need to mix two batches of NaOH to yield 4 L of 3 M NaOH.

Be sure students have read the lab and completed the pre-lab before going into the lab.
The lab requires two full days (in the lab) to complete.

Day 1) Students should be able to get through equation (3).
They must have added the NaOH to yield $Cu(OH)_2$
Heating the solution with a boiling chip is ideally where they should get to – boiling chips must be removed and not left in beaker over night.
If students are rushed for time and can't heat – don't worry, the reaction will proceed to completion on its own over night.

Day 2) Complete lab