Purpose

Gain familiarity with basic laboratory procedures, the chemistry of a typical transition element, and the concept of percent recovery

Learning Objectives

Identify and distinguish between different reaction types.

Calculate percent recovery using starting and final masses.

Laboratory Skills

Perform a decantation

Equipment

- Balance
- Beakers
- Stirring rod
- Evaporating dish
- Hot plate

Chemicals

- Copper wire
- Concentrated HNO_3
- 3.0 M NaOH
- 6.0 M H_2SO_4
- Granular zinc
- Concentrated HCl

Introduction

Most chemical syntheses involve the separation and purification of the desired product from unwanted contaminants. This activity uses two common separation techniques:

- **Sedimentation** - the process in which suspended particles settle out to the bottom of a container. Sedimentation is generally followed by decantation.
- **Decantation** - the liquid in a mixture containing solid particles is carefully poured off, retaining the solid particles in the original container.

This activity is designed to be a quantitative evaluation of students' individual laboratory skills in carrying out the above operations. At the same time, students will become acquainted with several fundamental types of chemical reactions:

- **Synthesis** - two or more reactants combine to form a more complex product. A generic synthesis equa-

tion is shown in Equation CC.1.

$$A + B \longrightarrow AB \qquad \text{(Equation CC.1)}$$

- **Decomposition** - a single reactant breaks down to form two or more products. Decomposition reactions are the reverse of synthesis reactions, as shown in Equation CC.2.

$$AB \longrightarrow A + B \qquad \text{(Equation CC.2)}$$

- **Single Replacement** - an element reacts with a compound to form a new compound and a different element. A generic single replacement equation is shown in Equation CC.3.

$$A + BC \longrightarrow AC + B \qquad \text{(Equation CC.3)}$$

- **Metathesis** or **Double Replacement** - two compounds interchange components to form two new compounds. This interchange is shown in the generic metathesis equation, Equation CC.4.

$$AB + CD \longrightarrow AD + CB \qquad \text{(Equation CC.4)}$$

Synthesis, decomposition, and single replacement reactions all involve the transfer of electrons and are also classified as **oxidation-reduction** reactions. Metathesis reactions occur without any transfer of electrons.

In this activity, students will carry out several chemical transformations involving copper and its compounds. The chemical reactions involved are the following:

$$Cu(s) + 4\,HNO_3(aq) \longrightarrow Cu(NO_3)_2(aq) + 2\,NO_2(g) + 2\,H_2O(l) \qquad \text{(Reaction CC.1)}$$

$$Cu(NO_3)_2(aq) + 2\,NaOH(aq) \longrightarrow Cu(OH)_2(s) + 2\,NaNO_3(aq) \qquad \text{(Reaction CC.2)}$$

$$Cu(OH)_2(s) \xrightarrow{\Delta} CuO(s) + H_2O(g) \qquad \text{(Reaction CC.3)}$$

$$CuO(s) + H_2SO_4(aq) \longrightarrow CuSO_4(aq) + H_2O(l) \qquad \text{(Reaction CC.4)}$$

$$CuSO_4(aq) + Zn(s) \longrightarrow ZnSO_4(aq) + Cu(s) \qquad \text{(Reaction CC.5)}$$

Reactions tend to proceed to completion whenever one of the products is removed from the solution or when a very stable substance is formed. The first reaction, Reaction CC.1, is an oxidation-reduction reaction. The copper metal is oxidized (loses electrons) to form copper ions while the nitric acid is reduced (gains electrons) to form nitrogen dioxide gas. This reaction is driven to completion by several factors, including the formation of nitrogen dioxide gas and the stable compound, water. The final reaction, Reaction CC.5, is also an oxidation-reduction reaction. In this reaction, zinc metal transfers electrons to copper ions to form zinc ions and copper metal. It

proceeds to completion because zinc is more readily oxidized than copper and, thus, zinc ions are more stable than copper ions. This reaction is also characterized as a single replacement reaction.

Both Reaction CC.2 and Reaction CC.4 are metathesis reactions. In Reaction CC.2, an insoluble precipitate is formed, driving this reaction to completion. Reaction CC.4 is a neutralization reaction in which the stable compound, water is formed.

Reaction CC.3 is a decomposition reaction. The triangle above the arrow indicates that this reaction is driven to completion by the addition of heat, which causes water to be removed from the reactant.

The objective of this activity is to recover the initial mass of copper with maximum efficiency. The percent recovery of the copper is expressed as the ratio of the recovered mass to initial mass, multiplied by 100, as shown in Equation CC.5:

$$\%\ \text{recovery} = \frac{\text{recovered mass of Cu}}{\text{initial mass of Cu}} \times 100 \qquad \text{(Equation CC.5)}$$

Procedure

Safety Precautions

Be careful not to get any of the nitric acid on yourself. If you do, wash it off immediately with copious amounts of water.

The NO_2 gas produced in the HNO_3 reaction is toxic.

This reaction must be performed in the fume hood.

1. Weigh ~0.1 g of copper pieces to the nearest 0.001 g and record its mass. Place it in a 250 mL beaker.

2. ***In the fume hood*** add ~1.5 mL of concentrated HNO_3 to the beaker. Describe the reaction as to color change and evolution of a gas. Gently heat the mixture on a hot plate to dissolve all copper metal pieces. After the reaction is complete, add 30 mL of deionized H_2O. What copper compound is present in the solution?

3. Add ~10 mL of 3.0 M NaOH to the solution in your beaker and describe the reaction.

4. Carefully heat the solution—while stirring with a stirring rod—just to the boiling point. Describe the reaction.

5. Allow the solid to settle, and then decant the supernatant liquid. Wash the solid with very hot DI water by adding a little water at a time, stir, then allow the solid to settle and decant. Repeat as many times as needed. What are you removing by the decantation?

6. Add 2-3 mL of 6.0 M H_2SO_4 to the solid. Describe the reaction. What copper compound is present in the solution now?

7. Add 0.5 g of granular zinc all at once and stir until the supernatant liquid is colorless. Describe the reaction. What compound is present in solution?

8. When gas evolution has become *very* slow, heat the solution gently (but do not boil) and allow it to cool. Decant the liquid. If zinc particles are still present, add a few mL of concentrated HCl to the beaker, let react and decant again. Repeat until all zinc is gone. What gas is formed in this reaction?

9. When gas evolution has ceased and the solution is colorless, decant the solution and transfer the precipitate to a pre-weighed porcelain evaporating dish.

10. Wash the precipitated copper with about 5 mL of distilled water, allow it to settle, decant the solution, and repeat the process. What are you removing by washing?

11. Prepare a steam bath as illustrated in Figure CC.1 and dry the product on your steam bath UNTIL DRYNESS. Describe the appearance of your recovered copper sample.

12. Allow the evaporating dish to cool. Then, wipe the bottom of the evaporating dish, weigh the evaporating dish plus copper, and record the mass. Calculate the final mass of copper.

13. Reheat the copper solid in the evaporting dis, cool and reweight. If there is no change in mass, then the copper solid is dry. If there is a change in mass, repeat this step for a third

Figure CC.1: Steam Bath

weighing.

14. Compare the recovered mass with your initial mass and use Equation CC.5 to calculate the percent recovery. Suggest possible sources of error in this activity.

15. Dispose of the chemicals in the designated receptacles.

1. Initial mass of copper (g) _____

2. Describe the reaction of Cu(s) and HNO_3(aq)

3. What compound is present in solution after the reaction with nitric acid? _____

4. Describe the reaction when adding NaOH(aq)

5. Describe the reaction when heating the solution

6. What are you removing by decanting? _____

7. Describe the reaction when adding H_2SO_4(aq)

8. What copper compound is present in solution after adding sulfuric acid? _____

9. Describe the reaction when adding Zn(s)

10. What compound is present in solution after adding zinc metal? _____

11. What gas is formed by adding HCl(aq)? _____

12. Mass of evaporating dish (g) _____

13. What are you removing by washing after the reaction with hydrochloric acid? _____

14. Describe the recovered copper

15. Mass of recovered copper and evaporating dish (g) _____

16. Mass of recovered copper (g) _____

17. Percent recovery (%) _____
Show calculations for percent recovery

18. Possible sources of error