Introduction to Natural Science 2006/07

Fall 2006 Quarter

Chemistry Lab III: "Chemical Reactions"

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This lab is adapted from
Gordon T. Yee, Jeannine E. Eddleton, & Cris E. Johnson, J. Chem. Educ., 81, 777, (2004)
And Stephen DeMeo, J. Chem. Educ. 72, 836, (1995)

Experiment 1
Copper Metal from Malachite Circa 4000 BCE

Introduction

The smelting of copper ore to obtain copper metal probably represents on of the big steps man made in his quest to utilize the natural resources in his environment for his benefit. In this experiment, we will mimic the steps the Copper Age Man might have used to produce copper metal from its ore. Our starting material will be the mineral malachite. Another copper containing mineral is azurite. Today however, azurite and malachite are more important as semiprecious stones used in jewelry than as ores of copper metal.

The procedure for isolating copper metal from malachite involves two steps:
Step 1: Roasting malachite to drive off carbon dioxide and water to produce copper(II) oxide.
Step 2: Reduction of the copper(II) oxide using carbon to produce copper metal.

Carbon (charcoal) is used in the second step to reduce copper(II) oxide to metallic copper. Charcoal is hardwood that has been heated in the absence of oxygen to drive off volatile organic compounds. As a result, it burns hotter and faster than wood. When charcoal is burned, a mixture of carbon monoxide and carbon dioxide are produced. Therefore it is very dangerous to burn charcoal indoors.

Pre-Lab Questions

Read the entire lab write-up three times.
Please show all work when doing calculations.

1. What is the chemical formula of malachite?
2. What it the color of malachite?
3. Write a balanced equation for the first step of the reaction (roasting malachite to form copper(II) oxide, carbon dioxide and water)
4. Write a balanced equation for the second step of the reaction (reaction of copper(II) oxide with charcoal to produce copper metal, carbon monoxide and carbon dioxide)

Experimental Procedure (work in groups of four)

Use the analytical balance only for weighing.

Step 1: Roasting malachite to produce copper(II) oxide

Obtain one malachite bead from your instructor. Clean and accurately weigh the sample to the nearest milligram in a weighing boat. Transfer the sample to a crucible and cover the crucible with its lid. Save the weighing boat to use again in the next step. In the hood, assemble a Bunsen burner and an iron ring on a ring stand and place an iron triangle on the ring (as demonstrated in class). Place the crucible in the triangle and adjust the height of the ring such that the tip of the inner blue cone of the Bunsen burner is touching the bottom of the crucible. **Using the blue flame of the burner**, heat the crucible for about 15 minutes to transform malachite to copper(II) oxide. **OBVIOUSLY, THE CRUCIBLE, THE METAL RING AND STAND GET VERY HOT**. Also, the flame sometimes can go out, so keep an eye on your burner. If the flame goes out, relight it, but remember that all surfaces will be hot. After the heating period, remove the heat and allow the crucible to cool for 15 minutes. Use tongs to handle the crucible. Remove the crucible from the stand and determine the mass of the copper(II) oxide product. Record the physical properties of your product.

Step 2: Reduction of the copper(II) oxide to produce copper metal
Add a little bit of charcoal powder to your crucible and then carefully transfer the cupper(II) oxide you made into the crucible. Add enough charcoal powder to cover the cupper(II) oxide. Gently tap the crucible once on the bench to help settle the contents. Cover the crucible and heat it on the Bunsen burner (same setup as before) for 90 minutes. Make sure that you are using the blue flame of the burner. Otherwise the reaction will not produce what you desire. (While this is heating, start on Experiment 2). Remove the flame and allow the covered crucible to cool for 15 minutes. Pour out the contents onto a clean, watch glass and isolate the copper metal using a spatula. Use a soft brush/clean paper towel to remove any charcoal stuck to your product. If it isn't shiny, trying wiping it on a paper towel to remove the thin layer of carbon attached to the surface. When you have cleaned your piece of copper, weigh and record the weight.

Allow the crucible to come to **room temperature.** Then transfer the **cool** crucible to the labeled nitric acid bath for cleaning (in the hood).

Characterization of the copper

- Record physical properties of your copper sample.
- Take a hammer and strike your copper sample a few times. Record observations. What property of copper is being demonstrated here? Is it a physical or a chemical property?
- Then take a piece of fine sandpaper or steel wool and polish the flattened surface. Record your observations. What property of copper is being demonstrated here? Is it a physical or a chemical property?
- If your sample is deep red, chalky, and falls apart, your sample is not copper metal. What could it be? Why were you unable to obtain pure copper? If this is the case, you need to continue the rest of the experiment with another group that had better luck producing copper from malachite.
- Set up a simple circuit with a battery, a light bulb and some wires. Use your piece of copper as a "switch" in this circuit. See if you could turn the light bulb on and off using your "copper switch". Draw a block diagram of your circuit in your lab notebook. What property of copper is being demonstrated here? Is it a physical or a chemical property? **Be sure to get your circuit checked by an instructor before you leave the lab.**

Post-lab questions

1. One possible side reaction of Step 2 is the reduction of copper(II) oxide to copper(I) oxide. What is the chemical formula of copper(I) oxide?

2. Copper(I) oxide is a red, non-metallic solid. Write a balanced equation for the production of Copper(I) oxide from copper(II) oxide and carbon. Another product of this reaction is carbon dioxide.

3. How could you deal with the production of Copper(I) oxide if you wanted Cu metal? (You can imagine that Copper Age Man had to address this problem).

4. Calculate the theoretical yield of copper.

5. Calculate a percent yield of copper.

6. How could you use density to verify that your final product is actually copper? Write down the procedure you would use to determine the density of your sample. What problems would arise and how would you go about overcoming them?

Experiment 2
Synthesis of zinc iodide from its elements

In this lab, we will synthesize zinc iodide from its elements, zinc and iodine.

Procedure: (work in pairs). Use the analytical balance for weighing.
Weigh accurately 2 g of iodine crystals and 2 g of granular zinc (10-50 zinc mesh). Record the weights accurately. Place both solids in a boiling tube and place in a test tube rack. Put the rack in the hood.

Take about 5 mL of de-ionized water into a small beaker and add 3 drops of 6M acetic acid. Label this as “acidic water”. Add this acidic water to the boiling tube containing the two solids (in the hood). Swirl to mix the contents.

Note: acidification of water is not required for the reaction between iodine and zinc to occur. However, slight acidity prevents the formation of zinc hydroxide, an undesired product in this reaction. Zinc hydroxide, a white solid, is produced when pH = 7 (neutral solution).

Observe the boiling tube carefully once the acidic water has been added. Continue to swirl the reaction mixture and **record all observations**. The reaction will take at least 10 minutes to complete. Note any color changes, gas bubbles, temperature changes etc.

Once the reaction is complete and the boiling tube has **reached room temperature**, (will be about 20 minutes) you should see some unreacted zinc at the bottom of the boiling tube.

Carefully **decant** the aqueous solution from the boiling tube into a **pre-weighed** test tube that contains a boiling chip **(weigh the test tube with the boiling chip)**, allowing the solid zinc to remain in the boiling tube.

Rinse the remaining solid zinc in the boiling tube with 1 mL of acidic water and slowly decant the liquid into the above pre-weighed test tube. Repeat with 2 more aliquots of 1 mL acidic water.

Add a small amount of de-ionized water into the boiling tube containing solid zinc. Wash the zinc and discard the liquid by decanting. Repeat this process a couple of times. Dry the zinc by heating the boiling tube with a Bunsen burner. When the zinc is completely dry, it will not stick to the walls of the boiling tube. Allow to cool to room temperature and accurately weigh the unreacted zinc.

Heat the test tube containing the liquid and the boiling chip **gently** over a Bunsen flame (**it is easy to lose your product during this step unless you heat gently**) till all the water evaporates (about 10 minutes). A white/off white solid should appear in the test tube. This is solid zinc iodide. Place the test tube in a desiccator to cool and dry. Weigh the tube.

Post Lab Questions:

1. What is the “limiting reactant” in this reaction? Justify your answer.

2. Calculate the mass of zinc that reacted with the amount of iodine.

3. Calculate the number of moles of iodine and the number of moles of zinc that reacted.

4. Using the molar ratios, determine the empirical formula of zinc iodide.

5. Use your data to verify that the law of conservation of mass holds true.

6. Calculate the theoretical yield of zinc iodide, the experimental yield, and the percentage yield.

7. Based on your percentage yield, would this be a good process to synthesize zinc iodide industrially? Why or why not?