THE STOICHIOMETRY OF ANCIENT METALLURGY

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Materials: Ring stand (2), tripod stand, clay triangle, small porcelain crucible with lid, Bunsen burner (2), malachite chips, granular ("fish bowl") charcoal, crucible tongs, forceps, striker, weighing boat

Ref: Yee, Gordon T.; Johnson, Chris E. *J. Chem. Educ.* **2004,** *81*, 1777-1779

Purpose: The purposes of this experiment are: (1) To prepare elemental copper through the techniques used by Stone Age men circa 3000 B.C.E; and (2) to trace the stoichiometry of a 2 step reaction through percent yield determinations at each step.

Learning Objectives:

1. Master the proper and most effective use of a Bunsen burner
2. Understand basic stoichiometry and be able to calculate the expected and % yield of each step of a reaction
3. Recognize and appreciate the historical aspects of metallurgy and apply them to a contemporary laboratory environment

Pre-lab: Complete the pre-lab questions **before lab.**

Discussion[1]: Technological advancements were for thousands of years the outcome of accidents. The greatest discoveries of the Stone Age period were driven not by a desire for scientific data, but rather by the activities that were part of everyday life for the ancient man. Stone Age men were drawn to naturally occurring materials that were aesthetically pleasing. They searched for strong, durable materials that could be used as tools. They also searched for materials that could be used as jewelry or as works of art. Around 3000 B.C.E, Stone Age men were drawn towards malachite, a bright green mineral that was abundant in the cliffs and hillsides that surrounded them. They wanted to use the appealing natural color of malachite for pottery and other works of art. While attempting to use malachite for such purposes, however, Stone Age men discovered that elemental copper (and copper alloys) were often produced under extreme conditions of heat and pressure. They soon realized the wide range of applications that copper and its alloys can be used for. The development of procedures to consistently produce copper, bronze, and brass led to a progression from the Stone Age to the Copper Age. The extraction of Copper from naturally occurring minerals was an enormous technological advancement that was mostly the result of an accident.

This lab is designed to mimic the metallurgical techniques of the Copper Age. Students will receive small chips of malachite, and through a 2 step reaction, will produce elemental copper through the production of CuO and its subsequent reduction to elemental Cu. The reaction will proceed as follows:

$$Cu_2CO_3(OH)_2(s) \rightarrow 2CuO(s) + CO_2(g) + H_2O(g)$$
$$2CuO(s) + C(s) \rightarrow 2Cu(s) + CO_2(g)$$

[1] http://www.uwgb.edu/dutchs/WestTech/xancient.htm

Students will only use materials that were available to the people of the Copper Age. Though this lab will connect students to the world of ancient metallurgy, it can also be applied to contemporary discussions on stoichiometry and oxidation/reduction chemistry. The expected product of each step of the reaction is shown below.

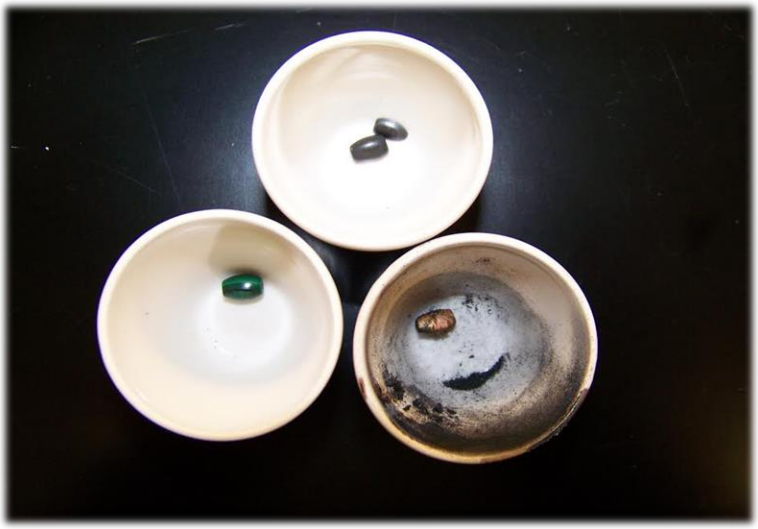


Figure 1: Malachite, Copper (II) oxide, and elemental Copper

Procedure: (Students will work in pairs for this exercise)

Part A: Crucible Preparation

1. Observe the correct usage of a Bunsen burner, as demonstrated by your instructor. Be able to create the sharpest cone of flame possible, as this is integral to the success of your experiment.

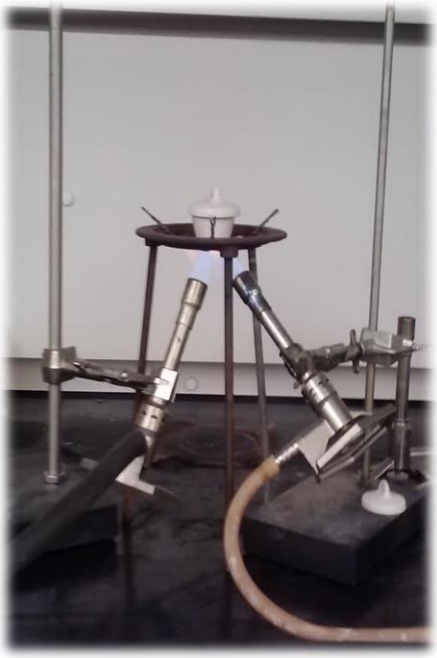
2. Obtain a small, clean porcelain crucible. It will be used throughout the entire experiment. Make sure your crucible has a cover that fits properly, as this is important for step one of the experiment.

3. Obtain 2 Bunsen burners and create the lab set up shown in Figure 2. Have your instructor aid you in this set up, as necessary.

IMPORTANT: Make sure the tip of the inner blue cone of flame is in contact with the bottom of your crucible. This is the hottest part of the flame and is required for the successful completion of the reaction.

Part B: Malachite Roast

Figure 2: Lab set up



1. Obtain 2 small chips of malachite. Measure the combined mass of the 2 chips and record this value in the data section.

2. Place your malachite chips in the crucible and heat them for 10 minutes using the setup shown in Figure 1. Make sure your crucible is covered to prevent the escape of your malachite chips. Again, ensure that the tip of each cone of flame is in contact with the bottom of the crucible. A strong red glow should be visible on the bottom of the crucible.

3. After 10 minutes, remove the crucible from the flames and allow it to cool for 5-10 minutes. Once the crucible is cooled to a safe temperature, weigh the product (CuO) and record this value in the data section.

Part C: Reduction

1. Obtain granular or "fish bowl" charcoal and add a small amount to the bottom of your reaction crucible. It should be enough to just cover the bottom surface of the crucible.

2. Place the CuO chips produced in Part B in the crucible.

3. Add more charcoal to the crucible until it is filled to within ¼ inch of the top. Your CuO chips should now be "sandwiched" between two layers of charcoal.

4. Heat your crucible for 30 minutes using the set up shown in Figure 2. It is not necessary to cover your crucible during this step. **Answer the in-lab questions during this time.**

5. After 30 minutes, remove the crucible from the flames and allow it to cool for 5-10 minutes. Once the crucible is cooled to a safe temperature, extract the product (elemental Cu) from the charcoal and weigh it. Record this value in the data section. **Note: If your product still has the gray color of CuO, re-apply the heat for an additional 5-10 minutes.**

Part D: Clean up

1. Place your extra charcoal into a container designated by your professor. It can be re-used for additional experiments.

2. Remove both Bunsen burners from their connection to the gas source, and put them away accordingly.

3. Clean your crucibles and remove all residues from within them.

4. Clean the surface of your lab bench. The copper product is yours to keep if you so desire.

Data Section:

	Mass (g)
Part A (malachite chips)	
Part B (CuO)	
Part C (elemental Cu)	

In-lab Questions:

1. Calculate the mass of copper present in your malachite chips.

_____moles

2. Calculate the expected mass of copper present in the CuO product produced in Part B.

_____moles

3. Calculate the actual mass of copper present in your CuO product from Part B.

_____moles

4. Calculate the percent yield for your malachite roast, based on moles of copper produced.

_____%

5. Aside from stoichiometric determinations, what other clues are there to tell you that you have created a new material in Part B?

Post-lab Questions:

1. Given the actual mass of the CuO produced in Step B, calculate the expected mass of elemental Cu that should be produced in Step C.

_____moles

2. Calculate the actual mass of copper produced in Step C.

_____moles

3. Calculate the percent yield for Step C, based on mass of Cu produced. Is this consistent?

_____%

4. Aside from stoichiometric determinations, what other clues are there to tell you that you have created a new material in Part B?

5. Did your reaction proceed as expected, in terms of stoichiometric consistency? If not, what were the sources of error that affected your experiment?

Pre-lab Questions

1. Watch the video at the following link to gain an appreciation for the metallurgical techniques of the Stone Age man.

http://www.bing.com/videos/search?q=ancient+malachite+to+copper&FORM=HDRSC3#view=detail&mid=57AA28A956C0D49DED4757AA28A956C0D49DED47

https://www.youtube.com/watch?v=_OrBw4L490Y

https://www.youtube.com/watch?v=8uHc4Hirexc

https://www.youtube.com/watch?v=l2FuvKTyRMQ

2. Determine the percent by mass of copper in malachite.

3. You are a caveman who harvests 2.65 grams of malachite from the local hillside. Given the reaction outlined in the Discussion section, how many moles of copper can you expect to harvest from your malachite? How many grams? A donkey can carry 40 kg over rough terrain. How much copper could be won from one donkey load of malachite?