ExPERIMENT 10

Empirical Formula Determination of Copper Sulfide

INTRODUCTION

The empirical formula of a compound indicates the relative numbers of atoms of each element in the molecule or formula unit. Three steps are required to determine a compound’s empirical formula:

1. Determine the mass of each element in a sample of the compound

2. Determine the number of moles of each element in the sample of the compound using the molar masses of each element

3. The empirical formula will be the smallest whole-number ratio for the moles of each element in the sample of the compound

Copper metal and powered sulfur can be converted to copper sulfide by strong heating. By measuring the mass of the copper before the reaction, and the mass of the copper sulfide after the reaction, the masses of the two elements composing the compound can be known. From these masses, the empirical formula of the compound can be determined. Because you will be ascertaining numerical data from a chemical reaction or for a chemical compound, this experiment is an example of quantitative analysis.

The container used to react the copper and sulfur will be a porcelain crucible. When the final product is produced in the crucible, the crucible is heated in the high temperature flame of the burner so that the crucible glows reddish-orange. This is called heating to red hot. The crucible and its contents are then allowed to cool, and weighed. The procedure of heating to red hot, cooling, and weighing is repeated until successive weighings are within 0.005 grams of one another. This assures that the product is pure and dry. This is called heating to a constant mass.
PROCEDURE

1. Students will work individually for this experiment. Except for the laboratory handout, remove all books, purses, and such items from the laboratory bench top, and placed them in the storage area by the front door. For laboratory experiments you should be wearing closed-toe shoes. Tie back long hair, and do not wear long, dangling jewelry or clothes with loose and baggy sleeves. Open your lab locker. Put on your safety goggles, your lab coat, and gloves.

2. Obtain a pair of crucible tongs from the back of the lab room.

3. Pick a clean, dry crucible and cover from your lab locker. Practice handling the crucible and the cover with the crucible tongs. Throughout the experiment, handle the crucible and cover only with crucible tongs, lintless paper, or gloves.

4. Using a milligram balance, determine the mass of the crucible and cover, and record its mass in your Data Table.

5. Obtain a 25 cm length of copper wire from the cart and roll it into a coil small enough to fit into the crucible, as shown below right. Place the coil into the crucible, replace the cover, and determine the mass. Record the mass of the crucible, cover and copper in your Data Table. You can now calculate the mass of the copper.

6. Obtain the powdered sulfur and transfer a small amount of it from its reagent bottle to a weighing cup or a glass or porcelain container by pouring.

**CAUTION:** Never place your microspatula or scoopula into a reagent bottle.

**NOTE:** If any sulfur is spilled on the lab bench, clean it up immediately, and dispose of it in the waste bottle in fume hood A. If there is any powder left on the lab bench at the end of the lab period, the instructor will deduct one point from everyone’s lab score as a charge for cleaning up after you.

With a scoopula, completely cover the copper wire in the crucible with powdered sulfur. Make sure there is no sulfur on the rim of the crucible. Place the cover on the crucible firmly.
7. Position the snorkel hood so that it is about 50 centimeters (20 inches) above the lab bench. Do so by loosening the three knobs and then adjusting the snorkel hood until it is in the position you want. Open the flow valve by turning it until it is parallel with the tubing, indicating that the vacuum is on.

8. Attach an iron ring to a ring stand, place a clay triangle on the iron ring, and set the crucible and cover in the clay triangle. The height of the iron ring should be adjusted so that the bottom of the crucible is about 5 cm above the burner. Place this apparatus under your snorkel hood, and adjust the snorkel hood so that it is about 30 cm above the crucible.
9. The crucible should be heated gently at first to reduce the risk of it cracking. Remove the burner from below the crucible and light it. Using a low, blue flame, hold the burner at a distance from the crucible and gently heat the crucible for a couple of minutes before placing the burner directly underneath it.

10. Once the burner is set below the crucible, gradually increase its heat output until it is finally heating so strongly that the bottom of the clay triangle or the bottom of the crucible turns red. At some point the sulfur will begin to burn.

   **CAUTION:** Sulfur burns with a pale blue flame that is hard to see and can cause severe burns.

   Continue to heat for several minutes after the last trace of burning sulfur disappears. Then, holding the burner in your hand, heat the entire outside surface of the crucible and cover to insure that all of the sulfur has burned off.

11. Allow the crucible, cover and contents to cool for 10 minutes. If a crucible is not cooled to room temperature when weighed, it will likely show a slightly lower mass due to the production of convection currents in the balance compartment. After cooling, determine the mass, and record the mass of the crucible, cover, and copper sulfide in your Data Table.

12. Reheat the crucible, cover and contents to red hot for 2 more minutes. Allow the crucible to cool for 10 minutes, and reweigh. If the mass after the reheating has a difference of more than 0.005 grams from the weighing before, reheat and weigh again until you obtain two consecutive weighings within 0.005 grams. When heating to a constant mass always use the last mass obtained for your calculations.

13. **The solid copper sulfide should be disposed of in the waste container in Fume Hood A.**

14. Clean and wipe dry your laboratory work area and all apparatus. When you have completed your lab report have the instructor inspect your working area. Once your working area has been checked your lab report can then be turned in to the instructor.
DATA TABLE

<table>
<thead>
<tr>
<th>Mass of Crucible, Cover</th>
<th>.</th>
<th>g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of Crucible, Cover, Copper</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>1 Mass of Copper</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>Mass of Crucible, Cover, Copper Sulfide</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>Mass of Crucible, Cover, Copper Sulfide</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>Mass of Crucible, Cover, Copper Sulfide (if necessary)</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>2 Mass of Copper Sulfide</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>3 Mass of Sulfur in Copper Sulfide</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>4 Percentage of Copper in Copper Sulfide</td>
<td>.</td>
<td>%</td>
</tr>
<tr>
<td>5 Percentage of Sulfur in Copper Sulfide</td>
<td>.</td>
<td>%</td>
</tr>
</tbody>
</table>

CALculATIONS

1.

2.
QUESTIONS

1. If all of the excess sulfur was not burned off when the copper sulfide was weighed, would the calculated percentage of copper in the copper sulfide be greater or less than the actual percentage? Explain based upon your calculation in Box 4.

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2. If all the copper did not react with the sulfur, would the calculated percentage of copper in the copper sulfide be greater or less than the actual percentage? Explain based upon your calculation in Box 4.

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3. Determine the empirical formula of the copper sulfide based on your calculated percentages of copper and sulfur in the compound.
4. A tin sample with a mass of 0.723 grams was reacted with oxygen, forming 0.918 grams of an oxide compound. Determine the empirical formula of tin oxide.

5. The compound oxydimethanol was determined experimentally to be composed of 30.8% carbon, 7.7% hydrogen, and 61.5% oxygen by mass. If oxydimethanol has a molar mass of 70 ± 10 g/mol, determine its empirical and molecular formula.