

Chemistry 122
SYNTHESIS OF
COPPER SULFATE PENTAHYDRATE

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Department of Chemistry
Western Washington University

INTRODUCTION

Synthesizing chemical compounds is an important and often fun part of chemistry. In this experiment, you will have the opportunity to synthesize some beautiful crystals of copper(II) sulfate pentahydrate. If you took Chemistry 121, you performed an experiment where you started with a piece of copper wire, and took it through a series of reactions, which produced a variety of copper compounds, ending with a reaction that regenerated solid copper metal. One of the reactions involved $\text{CuO}(s)$ and $\text{H}_2\text{SO}_4(aq)$ to produce $\text{CuSO}_4(aq)$ and water:



Your objective in this experiment is to synthesize non-aqueous copper(II) sulfate. As in the other experiment, one of the reactants is solid copper metal, in the form of copper mesh turnings. Here, you will carry out the chemical reaction shown below, in which the metallic copper is converted into solid crystals of copper(II) sulfate pentahydrate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.



Through this experiment, you will gain experience in handling chemicals and in using techniques for isolating a pure chemical compound. The calculations and post-lab questions will provide opportunities for you to work with chemical equations and perform calculations related to these equations which include determining the limiting reactant in the chemical equation, theoretical yield, and percent yield.

SAFETY PRECAUTIONS

You will be using a hot plate in this experiment. Hot plates are located on top of the hood at each lab station. When using a hot plate, take care to follow any heating instructions carefully. After you are finished with the hot plate, place the hot plate back on top of the hood to cool, but please do not wrap the cord around the hot plate – if the cord comes into contact with the hot surface, it will melt!

This experiment employs strong acids. For this reason, you are required to wear gloves while carrying out experimental procedures. Strong acids cause damage (immediate burns) upon contact with skin. If acid comes into contact with your skin, immediately rinse your skin with lots of water for at least 15 minutes and tell your instructor. Acids cause a “non-skid” feeling which may be accompanied by a burning sensation.

Wear departmentally approved eye protection at all times in the laboratory. Follow all additional laboratory rules and regulations provided by your instructor. Know the location and proper use of all laboratory safety equipment (safety showers, eye washers, fire extinguishers, etc.). Dispose of all chemicals in the proper waste containers located in the Waste Hood.

A material safety data sheet (MSDS) for each chemical used in this experiment is located in a binder in the lab. You should be familiar with the hazards associated with each chemical, as well as the instructions on safe handling and appropriate disposal. Your instructor will be available to assist you in interpreting this information

EXPERIMENTAL PROCEDURE

Before starting the experiment, read through the experiment and gather all of the necessary equipment. If you work carefully, you should only need to do one trial. *Record all observations on a separate sheet of paper. Thorough observations will help with the questions that follow your experimental work, so be specific.*

1. Tare your 250-mL beaker on the top-loading balance, and add approximately 3.0 grams of fine copper turnings. Record the mass of the copper to two decimal places in the data table on page 4. Place the beaker containing the copper inside a 600-mL beaker as secondary containment.
2. Using your 25-mL graduated cylinder, add 25 mL of 3 M sulfuric acid, H_2SO_4 , to the copper.
3. Take the beakers to the Working Hood and your instructor will add 7.5 mL of concentrated (16 M) nitric acid, HNO_3 . A brown gas, nitrogen dioxide, is produced in this reaction and is quite toxic, so leave the reaction in the Working Hood until the evolution of $\text{NO}_2(\text{g})$ has slowed.
4. Quickly transport your beakers to your bench-top hood. In your hood, heat the reaction mixture in the 250-mL beaker on a hot plate set to medium-low to medium for about 20 minutes or until all the copper has reacted away. (You must take the 250-mL beaker out of the 600-mL beaker for heating.)
5. Immediately after the copper has reacted away, quickly pour the hot solution into a 250-mL Erlenmeyer flask. (Note that the solution must be hot so that no crystals form in the beaker.) Let the solution in the flask cool on the bench-top for about 5 minutes, swirling occasionally.

If any unreacted copper remains in the reaction beaker after 20 minutes of heating, you will need to *decant* off the solution when pouring into the Erlenmeyer flask, being sure to leave the unreacted copper in the beaker. This copper should be rinsed into the waste container with a small amount of water.

6. Get a #6 rubber stopper, and once crystals begin to appear, stopper the flask tightly and run it under cold tap water until the solution is at room temperature. (Temperature of the solution can be estimated by feeling the outside of the flask.) Note that some NO_2 gas may form in the flask at this point, but it will not affect the crystallization.
7. Prepare an ice bath, which is a slurry of ice and water, in a 1-L beaker. Place the stoppered flask in the ice bath for 10-15 minutes, allowing it to sit undisturbed.
8. Use your 25-mL graduated cylinder to measure 20-mL portions of both 50% ethanol solution and pure (95%) ethanol into separate, labeled 50-mL beakers. Prepare two ice baths in larger beakers (i.e. 250-mL beakers), and place both beakers of ethanol on ice to cool.
9. Set up a filtering apparatus using your 125-mL filter flask, neoprene adapter, and Buchner funnel. (See the demo filtration apparatus set up in lab.) Insert a piece of filter paper into the Buchner funnel, and connect the side-arm of the filter flask to the vacuum using a piece of vacuum tubing. (Do not use the red tubing from your drawer.) Secure your filter flask to a ring stand with your utility clamp so that it doesn't tip over.

10. Turn on the vacuum all the way, and carefully, but quickly, pour the crystal solution onto the filter paper. Allow the vacuum to dry the crystals for a few minutes breaking up the clumped crystals with a spatula.
11. Add the 20 mL of ice-cold 50% ethanol solution to the Erlenmeyer flask and swirl to rinse and collect the remaining crystals in the flask. Quickly pour this solution over the crystals into the filter, and allow the vacuum to dry the crystals for 2-3 minutes. This step rinses out any crystals that remain in the flask and also washes the crystals in the funnel.
12. Rinse one final time with 20 mL ice-cold pure ethanol, pouring quickly so as to completely cover the crystals. Allow the vacuum to dry the crystals for five minutes.
13. Pre-weigh a watchglass on a top-loading balance, recording the mass to two decimal places in the table on page 4. Place the watchglass on a piece of paper, and carefully transfer the cake of crystals onto the watchglass. Dumping the crystals quickly helps to minimize loss of crystals. If any crystals fall onto the paper, simply pour them onto the watchglass. Weigh the watchglass and the crystals, recording the mass to two decimal places in the table on page 4. Calculate the mass of your final (wet) product), and record this mass in the table on page 4.
14. Have your instructor check your product, and if necessary carry out a second trial. Once your product has been approved, dump the crystals into the jar provided in the Waste Hood; do not add any water to the jar!!
15. Clean-up: Dump the waste from your filter flask into the waste container, rinsing with a small amount of water. Return all borrowed equipment: #6 stopper, 1-L beaker, vacuum tubing, spatula, and ring stand; be sure to keep your utility clamp.

CALCULATIONS & RESULTS

Due to the time constraints in lab, your final product is not completely dry. The ethanol washes and subsequent drying over vacuum do remove a significant amount of water from your product, but you would need to allow your product to sit overnight to get completely dry. In order to accommodate for the product being slightly wet, we are going to make the assumption that 10% of the mass of the wet product is due to residual water and ethanol. Therefore, be sure to report the “dry product” in the table on page 4 by taking this into account. You will use this mass in subsequent calculations.

Starting with the moles of reactants used in the reaction, develop and execute a strategy to calculate the percent yield you obtained in this experiment. You will need to determine the limiting reagent and the theoretical yield, based on the amount of the limiting reagent used in the reaction. Remember that the solid product has the formula $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$. The equation for percent yield is shown below:

$$\% \text{ yield} = \frac{\text{actual yield}}{\text{theoretical yield}} * 100$$

Use the following page to present your calculations and results in a **well organized manner**. Lab partners are encouraged to use their own format for their report. Be sure that all data and calculations are labeled, neatly presented, that units are given, that everything is expressed to the proper number of significant figures, and that *proper cancellation of units are clearly shown*.

Name _____

Lab Partner _____

Section _____

Date _____

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Data, Calculations and Results

Mass Data Table:

	Trial 1	Trial 2 (if needed)
Mass of Copper Turnings, g		
Mass of Watchglass + Product, g		
Mass of Watchglass, g		
Mass of Wet Product, g		
Mass of "Dry Product", g		

Calculations & Results:

