GOAL
In this experiment you will synthesize two compounds and gain experience with simple glassware and laboratory techniques.

INTRODUCTION
Most chemical experiments fall into one or more of the general categories of synthesis, characterization, and analysis. In synthesis experiments such as this week’s, chemists explore how to make new compounds. Chemists must determine not only what chemicals to mix, but also other factors such as the best conditions for reaction, and ways to separate a desired product from leftover starting materials or byproducts.

In Part I, you will prepare copper(II) oxide by reacting an aqueous solution of copper(II) sulfate with sodium hydroxide to give first copper(II) hydroxide and then the desired oxide product.

\[
\text{CuSO}_4(aq) + 2 \text{NaOH}(aq) \rightarrow \text{Cu(OH)}_2(s) + \text{Na}_2\text{SO}_4(aq)
\] Eqn 1

\[
\text{Cu(OH)}_2(s) \ + \text{heat} \rightarrow \text{CuO}(s) + \text{H}_2\text{O}(l)
\] Eqn 2

You will dissolve solid CuSO\(_4\)·5H\(_2\)O in water and then add aqueous NaOH to form copper(II) hydroxide. Since hydroxides tend to be gelatinous and thus don’t filter well, you will heat it to cause the Cu(OH)\(_2\) to dehydrate and form copper(II) oxide. See Eqns 1 and 2. Heating the product also causes very small precipitate particles to collect into larger particles in a process called digestion. As a result, we can use a fast-filtering coarse filter paper, rather than a slower, finer paper. You will use gravity filtration to isolate this product. Finally, you will rinse your product with some acetone. The acetone helps remove excess water. Because acetone is volatile, it will evaporate easily and help dry your product.

In Part II, you will prepare acetaminophen, a common analgesic sold under the trade name Tylenol. You will react p-aminophenol with acetic anhydride to form acetaminophen and acetic acid.

\[
\text{H}_{\text{C}}\text{C}\text{C}\text{N}\text{H} + \text{H}_3\text{C}\text{C}_{\text{O}}\text{C}_{\text{O}}\text{C}_{\text{CH}_3} \rightarrow \text{H}_{\text{C}}\text{C}\text{C}\text{N}\text{CH}_3 + \text{H}_3\text{C}\text{C}_{\text{O}}\text{H}
\] Eqn 3

p-aminophenol    acetic anhydride    acetaminophen    acetic acid

In lab, you will notice that the p-aminophenol, which should be white, is commonly steel gray. A small amount of highly colored impurity is present and discolors this reagent. The two reactants will be mixed
in a test tube along with some water. The test tube will then be heated in a boiling water bath to speed up the reaction. After heating, the mixture is cooled in an ice bath, which should cause the product to precipitate because it is less soluble at lower temperatures. Very often, however, a supersaturated solution forms. Scratching the inside of the test tube with a glass stirring rod should cause crystals to form because it will provide some nucleation sites for the crystals. You will use a Buchner funnel to isolate these crystals. Since acetaminophen is soluble in acetone, we can’t use it to wash the crystals this time. Instead, we will use a cold water wash to remove leftover starting materials and byproducts, without also dissolving our desired product.

PRE-LAB ASSIGNMENT
The structural formula for p-aminophenol is given in Eqn 3 at the bottom of Page 1. From this structure, we can determine that the molecular formula for p-aminophenol is C₆H₇NO. Use the structural formula for acetaminophen from Page 1 to find the molecular formula for acetaminophen. In your pre-lab notebook entry, copy both structures, and then give the molecular formulas and molar masses (a.k.a. molecular weights) of both compounds.

HAZARDS
Acetic anhydride has a strong and irritating odor. It should be dispensed in a fume hood. Avoid breathing its vapors, or allowing it to contact your skin. Sodium hydroxide is a strong base. The other chemicals used in this lab are not particularly hazardous. As always, avoid allowing chemicals to contact your skin. Rinse thoroughly with water if you accidentally spill anything on your skin or clothing.

LABORATORY DATA AND OBSERVATIONS
Be sure that you record what you do and the amounts of each chemical that you use in the left hand column of your notebook pages. Ensure that all data is clearly labeled. Record observations in the right hand column. The two parts of this experiment can be done in either order. Your instructor will assign half the class to start with each part.

PROCEDURE
Part I: Synthesis of copper(II) oxide, CuO
1. Weigh out about 0.8 grams of CuSO₄·5H₂O. Record the mass to 0.001 gram.

2. Transfer the solid to a 50 mL beaker and then add 10 mL of distilled water. Stir to dissolve.

3. In a separate beaker, get 10 mL of 2 M NaOH. Use a disposable pipet or eyedropper to add the NaOH solution dropwise to your copper solution. Use your glass stirring rod to stir the mixture. Be sure that you have been recording observations!

4. Set up your Bunsen burner. Position a ring stand so that the iron ring is 2-3 inches above the top of the burner. Add a piece of wire gauze and then place the beaker containing your reaction mixture on the gauze. Heat the mixture until the solution just begins to boil and the solid becomes uniform in color. Stir as you heat. Monitor your reaction carefully. Do not overheat.

5. Carefully set your beaker off the ring stand to cool slightly while you prepare your filter funnel. Fold a
piece of coarse filter paper into fourths. Open this into a cone and place it in your funnel. Support the funnel with an iron ring and place an empty beaker below the funnel. Use your wash bottle to moisten the filter paper so that it sticks to the sides of the funnel.

6. Pour your reaction mixture into your funnel, being sure that the liquid level stays below the top of the filter paper. Use a small amount of water from your wash bottle to rinse any precipitate that clings to your beaker into the funnel. While your mixture filters, take a small container to the reagent bench and get 3 mL of acetone from the dispenser.

7. After all the reaction solution has drained from your funnel, wash your precipitate with the acetone. To do this, use a disposable pipet to drizzle the acetone over the precipitate still remaining on the filter paper in your funnel.

8. When all the acetone has drained, carefully remove the filter paper from the funnel. Open the filter paper containing your solid product and set it on a watch glass. Use a marker to write your initials on the watch glass. Discard the liquid filtrate in the designated waste container.

9. Place the watch glass and filter paper in a 150°C oven for 20 minutes. While your product is drying, clean up your dirty glassware and weigh a clean watch glass. After 20 minutes remove your product from the oven and allow it to cool. Scrape your product off the filter paper and onto the weighed watch glass. Reweight. Some of your product will cling to the filter paper. Don’t worry about this. Record observations such as whether your product is dry, moist, flaky, crystalline, etc. Ask your instructor to visually inspect your product and initial your notebook. After it has been inspected, the solid may be discarded in the trash can. Be sure that you have recorded what you did and what you observed in your notebook.

Part II: Synthesis of acetaminophen

1. Weigh out 0.4 – 0.5 gram of p-aminophenol on a piece of weighing paper or plastic weighing boat, recording the mass to 0.001 grams. Transfer this solid into a 6-inch test tube, labeled with your name near its top.

2. Add 2 mL distilled water (measured with a graduated cylinder).

3. Take your test tube to the hood and add 0.5 mL of acetic anhydride from the dispenser. Give this mixture a quick stir with your glass stirring rod and then place your test tube in a boiling water bath on one of the hotplates. Once all of the solid has dissolved, continue heating in the boiling water for 10 minutes.

4. Remove your test tube from the water bath and allow it to cool briefly. Now prepare an ice bath. Fill one of your larger beakers with ice and add just enough water to make it slushy. Cool your test tube in the ice bath. Also put 5 mL of distilled water in a second test tube and cool this in the ice. If no crystals have precipitated from your reaction mixture within a few minutes, use your glass stirring rod to scratch the inside of the test tube until a few crystals begin to form. Return the test tube to the ice bath until it is thoroughly chilled and fully crystallized.
5. Set up a Buchner funnel, placing a piece of filter paper flat on the bottom of the funnel, as show to the right. Use your wash bottle to moisten the filter paper so that it sticks to the funnel. Turn the aspirator on full to create a vacuum. Use your stirring rod or a spatula to scrape your crystals from the test tube onto the filter paper. Release the pinch clamp on the aspirator trap to release the vacuum.

6. Wash your product twice with cold distilled water. To do this, add about half of your iced distilled water to the crystals in your funnel with the vacuum off. Briefly replace the pinch clamp to suck this water through the filter paper. Again release the vacuum and add the second half of the iced wash water. Replace the pinch clamp and allow the water to drain through.

7. Continue pulling air through the filter paper for several minutes to help your crystals dry. Be sure they are spread out in the funnel and not all in one clump. Release the vacuum, turn off the aspirator, and remove your filter paper from the funnel. Pour the liquid filtrate from the filter flask into a large beaker. Add a scoop of solid NaHCO₃. After the bubbling subsides, discard this solution down the drain with excess water.

8. Because time is too limited to allow your product to dry more naturally, blot your product between two layers of paper toweling to help speed drying. Weigh a clean watch glass. Scrape your product onto the watch glass and reweigh. Ask your instructor to visually inspect your product and initial your notebook. After it has been inspected, the solid may be discarded in the trash can. Be sure that you have recorded what you did and what you observed in your notebook.

RESULTS
For each part of the experiment, give the balanced chemical equation and calculate the theoretical and percent yields. Be sure to consider limiting reactants. You will need to look up the density of acetic anhydride for this calculation. You can find this information in the CRC Handbook of Chemistry and Physics, which is available electronically through the library and in the Trexler 472 computer room.

QUESTIONS
1. Percent yields over 100% indicate a problem. Very small percent yields are also unsatisfactory. Give one reasonable explanation for a yield that is too high and one reasonable explanation for a yield that is too low. Look over the procedure and see where things could have gone wrong.

2. In both parts of the experiment, you heated your mixtures but for different reasons. Describe the role of heat in each part of the experiment.

3. You blotted your acetaminophen crystals between two paper towels. This is not normally a preferred method for drying crystals. Based upon your experience, why not?

4. Your good friend at the College of Somewhere Else is taking Gen Chem and has written you for advice. She has prepared a compound that has precipitated from solution just as yours did in this
experiment. She writes, “Can I just let all the liquid evaporate and get the crystals that way? I have a bunch of different shaped funnels and some filter paper. Should I use these? Someone said I should wash the crystals. I don’t even know what that means.” Write several good paragraphs in response. Don’t just handle her questions in order. Instead, write a well-organized response that includes a description of the options for filtering and washing, including pros and cons of each option. Remember that you used two different types of funnels and two different washes in this experiment, so draw on your experience and information provided in the Introduction and Procedure sections of this experiment. Your answer will be graded for clarity as well as the correctness of its content. Remember that your audience is a Gen Chem student who seems quite lost.