Recrystallization

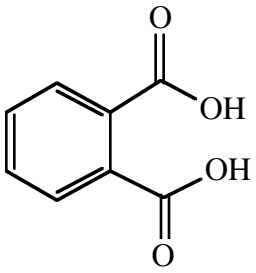
Introduction

When a reaction produces a solid product, most of the time that solid is not pure. So it will not have a narrow, reliable melting point and it will be difficult to characterize. Therefore, it will have to be purified. The most common way of purifying a solid is by recrystallization. This involves the following steps.

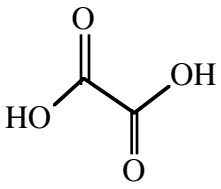
1. Dissolve the solid in a minimum amount of your boiling solvent to make a nearly saturated solution. For most solids, the solubility is proportional to the solvent's temperature.
2. Allow the solvent to **slowly** cool to room temperature, and then cool it in an ice-water bath.
3. Filter the newly crystallized solid through your Buchner or Hirsch funnel, using suction to speed up the filtering process.
4. Wash the crystals with a small amount of your **ice-cold** solvent.

When the boiling solvent cools slowly, the solubility of your solid will decrease to match the decrease of the temperature, and crystals will slowly form (I think this is one of the most beautiful things to watch in a chemistry lab!). Because the crystals form slowly, they will only select other compounds that perfectly fit into the growing crystal lattice. So only pure crystals will form, since impurities will not fit as well into the crystal lattice. After cooling all the way to 0 °C and quickly filtering the crystals (quickly so that the solvent does not warm up and re-dissolve your pure crystals), you should rinse your crystals with a small amount of **ice-cold** solvent. Think of this as being similar to the rinse cycle when washing your clothes. After the clothes have been washed in soapy water and the water has been drained, your clothes aren't clean. They're still coated with soapy water. So you need to rinse this off your clothes. The exact same thing is true with your crystals. They're coated with "dirty" solvent, and you need to rinse that solvent off. After the crystals have dried, then you can determine their purity by taking a melting point.

Experimental Procedure:



Phthalic Acid



oxalic acid

In the pictures above, remember that every corner is a carbon, and each carbon will have enough hydrogens on it to ensure that the carbon will have 4 bonds.

We will be recrystallizing impure samples of phthalic acid. The impurity is oxalic acid and it constitutes only about 5% of the mass of the sample. Oxalic acid is toxic and is found in the leaves of rhubarb, so you should only eat the stalks of rhubarb (unless you're one of those strange people who haven't discovered the subtle joys of this sour delicacy, and then you shouldn't eat any of it)!

Recrystallize 2.5 g of this mixture, using distilled water as your solvent. You should calculate the amount of boiling water needed by using the solubilities listed at the end of this handout. Then add about 10% more water because some of the water will evaporate as it reaches the boiling point. You should use an Erlenmeyer flask to do this recrystallization, and the amount of water should fill up

approximately 40-60% of the flask. If it is less than this range, the water will barely cover the bottom of the flask and will evaporate too quickly. If it is more than this range, you will run the risk of boiling over the top and spilling your solution.

Place the impure solid in the beaker and then add the water (you can add previously heated water to speed up the process) and a couple of boiling chips or a boiling stick. Boil the solution on a hot plate until all the solid dissolves. You may have to add a little water to completely dissolve the solid. After all of the solid has dissolved, remove the flask from the heat, place it on your bench top and allow it to cool to room temp. After it has cooled to room temp, place it in an ice-water bath. Different compounds crystallize at different rates, and our compound crystallizes fairly quickly. So it only needs to be in the ice-water bath for about 10-15 minutes. Filter the crystals by suction using the Buchner funnel. Moisten the filter paper with a small amount of water to make a good seal between the filter paper and the funnel. Scrape out any remaining crystals from your flask (use good spatula technique!) and add them to the pile in your Buchner funnel. Rinse your crystals with about 5-10 mL of **ice-cold** water (you can use this water to first wash any remaining crystals from your beaker before rinsing your collected crystals), and allow the pure crystals to air-dry.

After drying, record the weight and melting point of your pure product. You should also take the melting point of your original impure sample. Calculate the percent recovery of your phthalic acid (remember the starting mixture was only 95% phthalic acid by weight).

Solubility of phthalic acid in water

Temperature (˚C)	Solubility (g solute/100 mL water)
100	18
25	0.63
0	0.30

Questions (answers can be included in your discussion)

1. How do you choose a solvent for recrystallization? What solubility properties make a solvent a good choice for a recrystallization? What solubility properties make a solvent a bad choice for a recrystallization?

2. Why do you need to use ice-cold solvent when rinsing your purified crystals?

3. Do you expect oxalic acid to be more or less soluble in water than phthalic acid? Explain your answer.

4. Where will the oxalic acid be after your recrystallization is done?

5. How much of the phthalic acid should still be dissolved in the ice-cold water after it has cooled? Use this amount to calculate the best possible percent recovery.

6. An impatient and poorly prepared Stanford student decides to speed up the lab by taking her boiling hot solution and immediately placing it in an ice-cold bath, rather than allowing it to cool to room temperature first. How will this affect her results? Explain your answer.

7. Yet another poorly prepared and lazy Stanford student only gets a 45% recovery. List a couple of things he may have done incorrectly that would cause this poor yield (and **DON'T** list reasons like "he didn't scrape all the crystals off the side of the beaker).