

Recrystallization

Reference: Handout; Chemistry Lessons: recrystallization, solid purification, vacuum filtration, solubility; Green Lessons: benign solvent, recycle/reuse material; "Recrystallization" Movie; Zubrick, Ch. 13

Purpose: To use recrystallization techniques and purify benzoic acid

Table of Reagents:

Reagents	Amount	MW	Mmol	BP (°C)	MP (°C)	Density
Benzoic Acid	~2.5 g	122.12 g	20.49	249.2°C	121-125 °C	1.27 g/cm ³
Water (H ₂ O)	Varied	18.02 g		100 °C	0 °C	0.997 gm/cm ³

Safety:

- **Hot Plates**
 - Keep the surface of hot plates clean
 - Do not use excessive temperatures
 - Before putting hot plates away, unplug and let it cool down
 - Solvents can self-ignite on a hot plate
- **Benzoic Acid**
 - Could irritate upon skin and facial contact
- **Waste and Broken Glass**
 - Dispose lab waste in the appropriate waste container

Experimental Procedures	Data & Observations
1. Preheat ~100 mL distilled water in a 125 mL Erlenmeyer flask. Add ~2.5 g of crude benzoic acid and a boiling stone to a separate 125 mL Erlenmeyer flask. Record the mass of the crude benzoic acid used.	<u>Crude Benzoic Acid Used:</u> 2.4470 g <u>Hot Plate Temperature:</u> Set at 310 °C with the 100 mL of DI water <u>Observations:</u> Crude Benzoic Acid looks like black flakes
2. Add minimal amount of hot water to the crude benzoic acid (~50 mL of water). Mix by swirling and heat until solids are dissolved. Some insoluble purities may remain.	<u>Observations:</u> 50 mL of hot water added to benzoic acid. Some impurities remained (mostly small flakes).

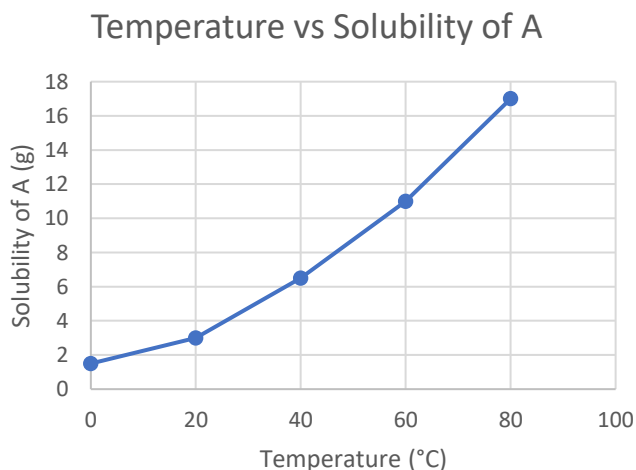
<p>3. Carefully remove the flask from the heat with tongs or a paper towel. Add a small amount of activated charcoal to the flask to remove the colored impurities. Swirl and bring the mixture back to a boil for several minutes.</p>	<p><u>Observations:</u> Mixture mostly black. Black tar forming at the side of the flask while it boils.</p>
<p>4. Prepare <u>Gravity Filtration Apparatus</u></p> <ol style="list-style-type: none"> Support stemless funnel in a ring Flute filter paper and place in funnel Place 125/250 mL Erlenmeyer flask below funnel Be sure to have ~50 mL of hot water available Pour a little bit of hot water into funnel to wet the filter 	
<p>5. Pour the crude benzoic acid mixture into the funnel to filter. Be sure to keep mixture at a constant, gentle boil to prevent crystallization. Rinse any remains of the mixture from the flask with hot water and pour in funnel.</p>	<p><u>Observations:</u> Crystals formed at the neck of the funnel. White crystals started to form.</p>
<p>6. Put filtrate aside to cool to room temperature (slow cooling process yields better crystals). When filtrate reaches room temperature, put in ice bath for ~10 min. ***If crystals don't form:</p> <ol style="list-style-type: none"> Scratch the flask at the air-liquid interface with glass rod. Create small ridges that increases surface area for crystallization "Seeding" solution with crystals of benzoic acid → triggers crystallization 	<p><u>Observations:</u> More crystals formed at the bottom of the flask.</p>
<p>7. Prepare Vacuum Filtration Apparatus</p> <ol style="list-style-type: none"> Clamp a 125/250 mL filter flask Place a neoprene adapter on the flask Connect the Buchner funnel on the neoprene adapter Place filter paper in the funnel Acquire another filter flask and connect it with a one-holed rubber stopper with glass tubing. 	

<p>f. Connect thick-walled vacuum tube from clamped filter flask to the glass tubing of the 2nd filter flask</p> <p>g. Take another vacuum tube and connect form 2nd filter flask to the vacuum</p>	
<p>8. Moist the paper with small amount of cold water. Turn on vacuum and filter benzoic acid mixture. Rinse crystals with ice, cold water. Dry and vent. Turn off vacuum and remove tube. Transfer crystals to clean watch glass to dry.</p>	<p><u>Watch Glass Weight:</u> 66.138 g</p> <p><u>Observations:</u> Pure benzoic acid looks powder-like as well as has a soft texture. Color → white</p>
<p>9. Crystal Dried. Record mass of pure benzoic acid and calculate % recovered from crude mixture. Measure the melting point. Dispose of the pure benzoic acid in the vial the TA has labeled. **These measurements will be taken next lab**</p>	<p><u>Mass of Pure Benzoic Acid:</u> 66.771 - 66.138 = 0.633 g</p> <p>% Recovered = $\frac{\text{Pure Product}}{\text{Crude Mass}} \times 100 =$ $\frac{0.633 \text{ g}}{2.4470 \text{ g}} \times 100 = 25.9\% \text{ recovered}$</p> <p><u>OptiMelt:</u> Start → 110 °C; Rate → 5 °C/min <u>Melting Point:</u> 123 °C</p> <p><u>Observations:</u> Sample melted at the same range benzoic acid melted, which means our sample is pure benzoic acid.</p>

Post-lab Questions:

1. a.

Temperature (°C)	Solubility of A in 100 mL of water(g)
0	1.5
20	3.0
40	6.5
60	11.0
80	17.0



b. Mixing 10 g of A and 100 mL of water, and afterwards heating it to 80 °C will result in all of A being dissolved. This is shown in the table in which the max amount of A being soluble in 100 mL of water is 17 g, concluding that 10 g will be dissolved.

c. In a situation in which the solution is cooled, precipitate will begin to form between 40 °C – 60 °C. This is because 6.5 g of A dissolves at 40 °C and that 11 g of A dissolves at 60 °C, which means if we start to cool the compound A to reach those ranges, the compound will start to precipitate.

d. Supposed the 10 g of A in the solution is cooled to 0 °C, then only 8.5 grams will precipitate from the solution (10 grams – 1.5 grams = 8.5 grams). The percent recovered of A is

$$\begin{aligned}\% \text{ Recovered} &= \frac{\text{mass recovered material (purified compound)}}{\text{mass of starting mixture (impure compound)}} \times 100 = \\ &= \frac{8.5}{10} \times 100 = 85\% \text{ recovered}\end{aligned}$$

2. a) My percent recovery for benzoic acid is 25.9%.

$$\% \text{ Recovered} = \frac{\text{Pure Product}}{\text{Crude Mass}} \times 100 = \frac{0.633 \text{ g}}{2.4470 \text{ g}} \times 100 = 25.9\% \text{ recovered}$$

b) Our low percent recovery tells us that most of our crude material was lost during the experiment. In addition, it is impossible to obtain 100% recovery in a recrystallization since each time your filtrate, small undissolved pieces of solid benzoic acid might not have filtrated through. Also, during crystallization, some of the benzoic acid might not have crystallized in the cooling process resulting in more benzoic acid being lost during the vacuum filtration.

3. Our purified benzoic acid melted at 123 °C. This result shows that our recrystallized product is pure benzoic acid, since our purified benzoic acid did not exhibit melting point depression as well as the melting point for our purified benzoic acid falls within the melting range of benzoic acid (121 – 125 °C).

4. Characteristics that makes a good recrystallization solvent is that it can dissolve the compound while its hot, however, it cannot dissolve the compound while its cold. Water is a good choice of a solvent for benzoic acid as it cannot dissolve benzoic acid at room temperatures, while impurities would be able to dissolve in the water at room temperatures. In addition, as part of the properties of a good recrystallization solvent, water dissolves benzoic acid while its hot, but does not dissolve it while its cold.

5. One reason why it is not a good idea to crank up the heat on the hotplate is that solvents can self-ignite on the hot plate, which can possibly harm you as well as the people around you. Another reason why it is not a good idea to crank up the heat is that excess heat can actually denature organic compounds possibly resulting in a lesser percent recovery at the end. (Also, a waste of electricity)

6. The use of a stemless funnel instead of a stemmed funnel during the gravity filtration step is necessary, since as the solution travels down the “stemmed” funnel, some benzoic acid might have already crystallized on the funnel resulting in loss of the purified compound as well as making it harder for material to travel down the funnel. In the use of a stemless funnel, this loss of purified compound and premature crystallization is terminated.

7. One thing to do in inducing crystallization of a supersaturated solution is by scratching the inside of the Erlenmeyer flask with a glass rod. This results in creating small ridges that increases the surface area for crystallization. Also “seeding” the solution with a crystal of benzoic acid will do a similar effect.