

CHEM-333: EXPERIMENT 2: EXTRACTION:

Prelab Assignment: Read chapter 4.

In this lab you will perform an extraction (Chapter 4; Experiment B).

Extraction is one of the easiest purification methods in the organic chemist's tool kit. The method exploits the different solubilities of compounds in immiscible (forms two layers) solvent mixtures. Here we will take advantage of the observation that many organic acids are insoluble in neutral/acidic water but are soluble in basic water. Follow the protocol and make sure that you vent your funnel.

Overview:

This experiment involves the separation of a combination of the neutral and acidic compounds listed below;

<i>Acids</i>	<i>Neutral</i>
<i>p</i> -Toluic acid	benzil
Benzoic acid	fluorene

The final identification will be by melting point.

Dissolve 0.5 g of your unknown in 25 ml of ether and place it in a **125 ml** separatory funnel. Extract the ethereal solution with 5% aqueous NaOH solution (3 x 15 ml). Combine the aqueous NaOH extractions and back-extract them with ether (15 ml). Combine the ether extracts and dry them over anhydrous Na₂SO₄, filter and remove the solvent on the rotary evaporator to give the neutral compound. Air dry the compound and take a melting point. If the melting point is not very sharp, recrystallize using ether/hexane.

To obtain the acid component of the mixture, cool the combined NaOH extractions to 0 °C using an ice bath and carefully acidify with 10% HCl solution, swirling gently after each addition (NB: Check the pH using indicator paper). Pour the mixture into a **125 ml** separatory funnel and extract with ether (3 x 15 ml). Combine the ether layers and dry over anhydrous Na₂SO₄, filter and remove the solvent on the rotary evaporator to afford the acidic component. Air dry the product and obtain a melting point as above. If the melting point is not very sharp, recrystallize using ether/hexane.

1. **Care!** Remember to *vent* the separatory funnel frequently to avoid the build-up of pressure.
 - a. Be sure to point the separatory funnel away from everyone when venting.
 - b. Before adding your solution to the funnel make sure the stopcock is closed
 - c. Before you attempt to drain the sepfunnel, remove the stopper.
2. Use a ring stand to support the **125 ml separatory funnel**.
3. Wash glassware before returning it to drawer.
4. Wash any chemicals spilled on yourself immediately.
5. If the isolated substance has a low melting point, the sample may need to be recrystallized in order to obtain a sharp melting point. *See instructor for details.*
6. Waste liquid goes in waste jug, capillary tubes go in marked jar and filter paper waste is in a separate container. **Do not put anything down the sink.**

Common separatory funnel mistakes:

1. **Vent!** When mixed in a closed system, many solvent mixtures build up pressure. Vent the funnel by firmly holding the stopper on, inverting the funnel and opening the stopcock making sure that you do not point it towards anyone.
2. **Remove the stopper:** As soon as you are done shaking and venting, take the stopper off to prevent pressure buildup. You will also find that the funnel will not drain properly (if at all) when the stopper is on.
3. Before you start using your funnel make sure the ring is appropriate for supporting your funnel. This sounds duh-obvious but you would be surprised how many people have broken funnels by sending them straight through a ring that's too big. (No its not worth two points and your out a \$45 funnel!)