

# Experiment 25

## Recrystallization of an Unknown: Choice of Solvent

**Reading:** Handbook for Organic Chemistry Lab, section on Recrystallization (Chapter 12).

In this laboratory period you will purify an unknown solid by recrystallization. Your TA will assign to you one of the “recrystallization unknowns” (#1–8). In order to choose a suitable recrystallization solvent (or solvent pair) for your unknown, it will be necessary to carry out solubility tests as outlined in the “How to Choose a Recrystallization Solvent” of the Crystallization section of the *Handbook for Organic Chemistry*. The section entitled “Procedure for Determining a Recrystallization Solvent” will be especially helpful.

When you have determined which solvent is the best for the recrystallization of your unknown, weigh out an amount of the unknown (about 0.5 g), recrystallize it, and weigh the recrystallized unknown in order to determine your percent recovery. Take a melting point of the unknown; this should give you a fairly good idea which compound it is. To confirm your guess, take a mixed melting point of your compound with the pure compound.

If none of these single solvents proves suitable for recrystallizing your unknown, you will need to investigate the use of a solvent pair, as described in the *Handbook*.

The solvents that will be available to you for testing are:

<i>Solvent</i>	<i>Boiling Point</i>	<i>Density (g/mL)</i>
Methanol	65 °C	0.791
Ethanol, 95%	78 °C	0.789
Acetone	56 °C	0.792
Distilled Water	100 °C	1.00
Petroleum Ether	35-60°C	~0.650
Hexanes	69 °C	0.654

The unknown that you will receive will be one of the compounds shown in Figure 25-1.

### **Safety Precautions**

All the solvents used in this lab are mildly hazardous and most are flammable so exercise due care in handling them. Wear your gloves and protective clothing.

### **Wastes**

*Organic Waste:* All used recrystallization solvents.

*Solid Waste:* Recrystallized unknowns.

## Experiment 25: Recrystallization of an Unknown

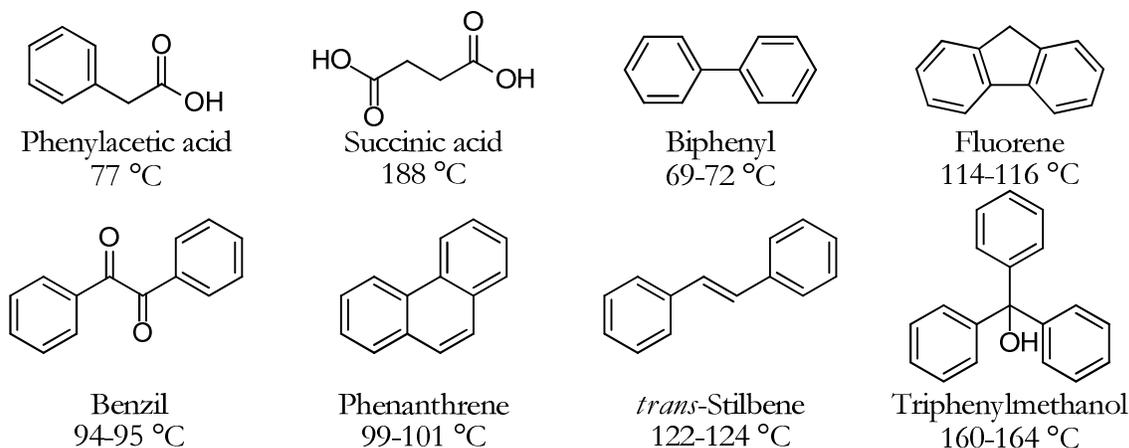


Figure 25-1: The possible compounds that you may be assigned for this experiment.

### Lab Report

Your conclusions should include:

- How efficient was your recrystallization? What could you have changed to improve its efficiency?
- Which of the unknown compounds were you given? How do you know?
- What was your percent recovery?

### Study Questions

- 1) Could the following solvent pairs be used for recrystallization? Explain why or why not.
  - a. Hexanes and water.
  - b. Chloroform and diethyl ether.
  - c. Propanol and ethanol.
- 2) For each of the solvents listed below, indicate an advantage and a disadvantage in their use as solvents for recrystallization.
  - a. Water.
  - b. Methanol.
  - c. Benzene.
  - d. Carbon tetrachloride.
  - e. Acetone.
- 3) A student crystallized a compound from benzene and observed only a few crystals when the solution cooled to room temperature. To increase the yield of crystals, the student chilled the mixture in an ice-water bath. The chilling greatly increased the quantity of solid material in the flask. Yet, when the student filtered these crystals with vacuum, only a few crystals remained on the filter paper. Explain this student's observations.