

RECRYSTALLIZATION OF BENZOIC ACID

Required prelab readings: Mohrig Sections 6.1 (pp 47); 9.1-9.6 (pp 100 – 111) and 9.9 (114-116).

Previous techniques you must understand and be able to perform: Melting points.

Recrystallization is the first of four methods of purification you will be learning this term (recrystallization, distillation, chromatography and extraction). Recrystallization is probably the most important method for the purification of solids since it can be used in conjunction with other types of purification. Although recrystallization can be performed on virtually any amount of material, one significant advantage of this technique is that it can be performed on very large scale. Kilogram quantities of material can be purified by recrystallization. Purification of that much solid material by other techniques, chromatography for example, would be impractical. It is of utmost importance to learn this technique well since you will use it throughout the year of Organic Chemistry Lab and later in Inorganic Chemistry Lab.

Procedure:

Weigh out a 1.0 g sample of impure benzoic acid for recrystallization and place it in a 125 mL Erlenmeyer flask. Be certain to record your unknown letter in your lab note book. The water used for experiments in our labs is purified by a technique known as reverse osmosis (RO). The purity of RO-water is equal to or better than that of distilled water. The RO-water comes from the grey taps in the labs throughout the building. Add about 10 mL of water to your benzoic acid and place the benzoic acid/hot water mixture on the hot plate and bring to boiling (or close to boiling). If the benzoic acid has not completely dissolved after 15 – 30 seconds add water (in approximately 5 mL increments) as needed until all of the benzoic acid has dissolved. Be cautious with the amount of water since there is an insoluble material present. YOU will have to decide when all of the benzoic acid has dissolved. Total the volume of water you have added and add an additional 25%.

An important point about all lab procedures: READ AHEAD. Don't find yourself finishing a step then reading the following: "Now immediately perform the following step", when the equipment and reagents for the following step are nowhere to be found. As it applies to today's experiment you should have the gravity filtration apparatus (see below) set up close to the hot plate before you begin heating your benzoic acid/water mixtures.

Gravity filter the hot solution through a wide neck funnel and allow the filtrate to stand. Rinse the Erlenmeyer with a small amount of boiling water. After cooling to room temperature, and a final cooling in an ice bath (**CLAMPED**), suction filter the purified benzoic acid using a small Büchner funnel attached to a clamped filter flask; the flask is attached *via* rubber tubing directly to the hood's vacuum line – a trap (fig. 9.5, p. 110) is not needed; be sure to wet the filter paper with ice-cold water before pouring your crystals. Rinse the crystals with a minimum of ice-cold water. Continue to pull a vacuum on the funnel for 1-2 minutes. Air dry your sample to constant weight and take a melting point. You may find it necessary to let your sample dry in your drawer until the next lab period before taking a final weight and melting point. Have the instructor record and initial the weight of your recovered material in your lab notebook. After weighing, place your purified product in the appropriately labeled container for re-use.

The General Recrystallization Technique:

After an appropriate solvent (or solvent combination) is determined by trial and error, the solute is dissolved in a minimum amount of the solvent at or near the solvent's boiling point. If undissolved impurities are present the mixture is gravity-filtered hot to remove the impurities. The filtrate is cooled (usually to 0 °C), sometimes scratched with a glass stirring rod to induce crystallization, and the crystals that form are collected by suction filtration and washed with small amounts of cold recrystallization solvent.

**RECRYSTALLIZATION OF BENZOIC ACID
DATA SHEET**

NAME _____ **Section** _____

Unkown letter: _____

Initial mass _____

Mass of recovered benzoic acid: _____

Percent recovery _____

Melting point: observed: _____

literature: _____

literature source: _____

Percent Recovery calculation: _____

Structure of benzoic acid

Answer the following questions:

1. What is the purpose of recrystallization?

2. What types of organic compounds are soluble in water, insoluble? Give specific examples of each with structures.

3. The solubility of a compound is 59 g per 100 mL in boiling methanol, 30 g per 100 mL in cold methanol, whereas its solubility in water is 7.2 g per 100 mL at 95 °C and 0.22 g per 100 mL at 2 °C. Which solvent would be better for the recrystallization of the compound? Explain.
4. What explanation can you give for the fact that benzoic acid is soluble in hot water and not soluble in cold water? Answer your question at the molecular level discussing the specific molecular interactions involved.