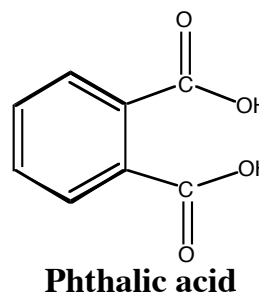


Experiment 1: Recrystallization

Read pp 183-193 and 195-197, Technique 15, in *TOC*. The theory and technique of the purification of solid compounds by recrystallization is fully explained in these pages. View the video: 5.3 Recrystallization (Macroscale), which can be found on the web at bcs.whfreeman.com/mohrig2e/ under "Techniques in Organic Chemistry Movies and Spectra". Under "Select your glassware type", choose **Williamson**. Read also pp 49-50 (6.1) to learn about boiling stones and sticks, and page 52 to review the use of hot plates for heating flat-bottomed containers.

Part 1. Recrystallization of Pure Phthalic Acid

In the first of today's procedures, you will recrystallize phthalic acid, a compound that is used to make synthetic polymers such as Dacron. Phthalic acid is very soluble in boiling water, **18 g/100 mL**, and is much less soluble in chilled (14°C) water, **0.54 g/100 mL**. Thus, water is a very good recrystallization solvent for phthalic acid.



Outline the steps of the following procedure. As part of your pre-lab assignment, calculate the volume of boiling water required to dissolve 1 g of phthalic acid (use the data given above).

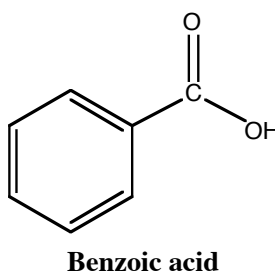
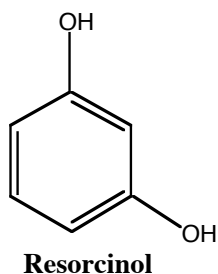
Place 1.0 g of phthalic acid in a 25 or 50 mL Erlenmeyer flask. Place 10 mL of water in a graduated cylinder. Add 4-5 mL of the water to the solid in the flask along with a wooden boiling stick, then begin heating the mixture on a hot plate. As soon as the water begins to boil, continue to add water dropwise from the graduated cylinder until the solid *completely* dissolves. If 10 mL of water has been added and the solid still has not completely dissolved, refill the graduated cylinder, and continue to add water, but at a faster rate. Record the total volume of water required to dissolve the crystals. Take the flask off the hot plate, and remove the boiling stick. Allow the solution to cool undisturbed to room temperature. During this slow cooling, crystals of the solid will form. When the flask has reached room temperature, place it in an ice bath for at least 10 minutes.

Set up a vacuum filtration apparatus using the small Buchner funnel and 125 mL side-arm flask from your drawer (see p. 192 in *TOC* and the [Figure Page, Expt. 1](#)). Place a disk of filter paper in the funnel and collect the crystals by vacuum filtration. You may use a spatula to assist in transferring the crystals to the funnel, then wash the phthalic acid in the funnel with small amounts of ice cold water. Transfer the crystals from the funnel to a watchglass, place another piece of filter paper on top of the crystals and press firmly to remove some of the water. Leave them in your drawer to dry until your next lab period. You will obtain the weight of your recovered product next week. Two days after your next lab period, you will be required to hand in the Results to your TA. The filtrate in the side-arm flask may be poured into the sink.

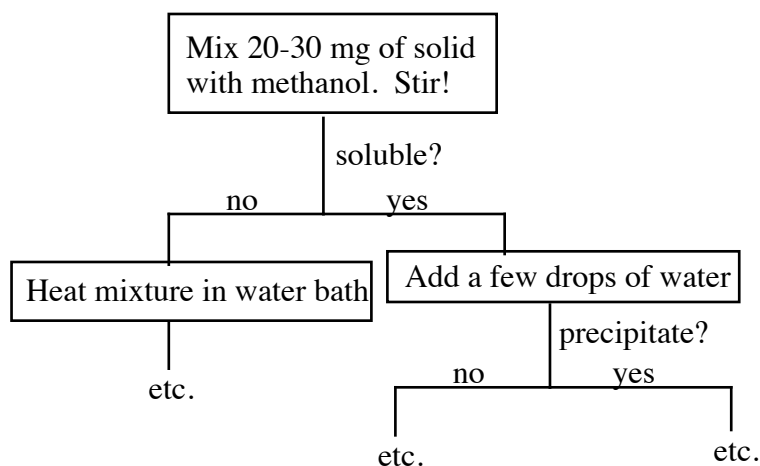
Part 2. Solubility Tests

In the second part of today's experiment, you will test the solubility properties of resorcinol and benzoic acid (see top of page 2) in seven different solvents: methanol, ethanol, acetone, acetic acid, water, toluene and ligroin (also known as petroleum ether). See *TOC*, Section 15.3, and the table on page 185. Knowing the solubility properties,

you will be able to draw some conclusions regarding an appropriate recrystallization solvent or solvents for each compound.



Use a flow chart format to **outline** the steps of the following procedure (see below for an example). You should perform these tests while your phthalic acid is crystallizing.



Caution! Methanol, ethanol, acetone, toluene and ligroin are flammable!

To test the solubility of a solid, transfer a "spatula-tip" full of the compound (you may weigh out 20-30 mg of one of the solids to get an idea of how much should be used) into a 10x75 mm test tube and add 10 drops of solvent. Stir with a thin stirring rod, break up any lumps, observe and record whether or not the solid is readily soluble at room temperature.

If the solid does not dissolve in a given solvent at room temperature, heat the test tube containing the mixture in a beaker of water heated on a hot plate. If the solid completely dissolves at elevated temperature, record that it is soluble in the hot solvent. If some but not all dissolves, record that it is slightly soluble, and continue to add drops of solvent until dissolution is complete. Once the substance has been dissolved in hot solvent, allow the solution to cool to room temperature, then stick the test tube in ice. If necessary, induce crystallization by rubbing the walls of the tube with a stirring rod. Describe the nature of the crystals which form (for example, needles, plates or blocks). If

the solid remains undissolved even at elevated temperature, simply record it to be insoluble in both the hot and cold solvent.

If the solid dissolves in methanol, ethanol, acetone or acetic acid at room temperature, add a few drops of water to the solution to see if the solid precipitates. If it does, heat the mixture in the water bath and observe if the solid re-dissolves. Adjust the composition by adding drops of solvent and/or water as necessary until the solid has *just* dissolved and the solution is saturated at the boiling point. Allow the solution to cool to room temperature, then stick the test tube in ice. Describe the shape of the crystals which form. Do not add water to either toluene or ligroin since it is not miscible with these solvents.

Pour the mixtures contained in your test tubes into the **Laboratory Byproducts** jar.

Notes:

- Acetic acid will freeze when placed in ice. Do not interpret frozen acetic acid as crystal formation.
- Ligroin will evaporate quickly when placed in boiling water. Cool down your water bath (by adding ice) before heating a test tube that contains ligroin.

Prepare a table in your notebook *before* coming to lab in which you will record your observations. You will need to record the solubility at room temperature, the solubility at elevated temperature, the result of adding water to a solution of the compound (when applicable) and the appearance of the crystals.

In order to write a complete discussion for Assignment 1, you will need to use the Spartan software that is on the computers located in the support room. Bring the [Molecular Modeling PDF](#) document to lab with you, both for this experiment and for Experiment 2. Find time during these two lab periods to carry out this computer work on the molecules used in Experiment 1, Part 2. Solubility Tests. Your TA will assist you, and you may work on it with other students.

Name _____ Date _____

T. A. _____ Lab period _____

Results and Calculations (to be handed in two days after the next lab period)

Calculate the percent recovery of dry, recrystallized phthalic acid (show your work).

Calculate how many grams of phthalic acid remained in the chilled water (the "mother liquor") based on the solubility data given in the introduction (show your work).