Recrystallization

Part II. Selecting an Appropriate Solvent

In this experiment, you will test the solubility of resorcinol (also called benzene-1,3-diol) and benzoic acid in four different solvents: water, methanol, acetone, and toluene. After observing the solubility properties, you will be able to identify the most appropriate recrystallization solvents for these compounds.

Resorcinol is used as an antiseptic and disinfectant and in the synthesis of dyes and plasticizers. Benzoic acid, along with its salts, is used as food preservative.

HO\(\begin{array}{c} \text{resorcinol} \\
\text{benzoic acid}
\end{array}\)

General Procedure

Caution! Methanol, acetone, and toluene are flammable! No flames!

Obtain 8 clean, dry 10x75 mm test tubes. Label each tube with a different solvent (water, methanol, acetone, and toluene). To each of four test tubes, transfer 15-20 mg of the first compound. (It is only necessary to weigh the sample for the first tube, then use that as a visual guide for the other three.) Add 10 drops of each solvent to the tubes. Swirl to mix well. Only if necessary, use a thin stirring rod to break up any lumps. Observe and record whether the solid is readily soluble (“s”) or insoluble (“i”) 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 hot (~80-90°C) water bath (in a beaker) heated on a hot plate. [NOTE: Acetone will evaporate quickly when placed in hot water (see boiling points). Cool down your water bath (by adding ice) before heating a test tube that contains this solvent.]
  - If the solid completely dissolves at the elevated temperature, record that it is soluble in the hot solvent. Record the temperature.
  - If some but not all dissolves, record that it is partially soluble (“ps”) and slowly add drops of hot solvent until it completely dissolves. Record the number of drops you add. Record the temperature.
  - Once the substance dissolves in hot solvent, allow the solution to cool completely to room temperature, and then place in an ice bath for about 10 minutes. If necessary, induce crystallization by rubbing the walls of the tube with a stirring rod. Describe the shape of the crystals that form (needles, plates, or blocks, etc.).
    - If the solid remains undissolved, even with additional drops of solvent added at the elevated temperature, simply record it to be insoluble in the hot solvent.

- If the solid dissolves (“s”) in methanol or acetone at room temperature, add a few drops of water to the solution to see if the solid precipitates. [NOTE: Do not add water to toluene since they are not miscible.]
  - If no precipitate forms, record that it is readily soluble at room temperature in the solvent/water mixture.
  - If a precipitate forms, record that it is partially soluble at room temperature in the
solvent/water mixture. Cool the mixture in ice bath for about 10 minutes. Describe the shape of any crystals that form.

Repeat the solubility tests in the next four test tubes with the second compound.

Pour all mixtures from your test tubes into the Organic Waste jar in the hood. Rinse the tubes sparingly with acetone and wash them at the sink.

Record all your results in a table in your notebook with the following information for each compound in each solvent: the solubility at room temperature, the solubility at the elevated temperature (if applicable), the solubility at room temperature-with water added (if applicable), and the appearance of the reformed crystals (if applicable). Include details such as temperature, and number of drops added.

You may find it useful to outline the procedure and/or the results in a flowchart format as shown below.

[Part II adapted from Undergraduate Organic Chemistry I, Boston College.]

Follow-up Questions for Recrystallization Parts I and II
1. Why must boiling chips be used?
2. Why is cold solvent used for washing phthalic acid crystals collected in the filter?
3. Where will the oxalic acid be after your recrystallization is complete?
   Also, answer questions 1, 2, and 4 on page 235 of TOC, 4th ed (page 197 of TOC, 3rd ed).

Conclusion for Recrystallization Parts I and II: Summarize your results. Report the purity and percent recovery of phthalic acid. Identify which solvent(s) would be most appropriate for recrystallizing resorcinol and benzoic acid. Justify your reasoning. Cite any important considerations that should be observed when performing a recrystallization.