7.6. Two-Solvent Recrystallization Guide

Overview:

For a two-solvent recrystallization, you should have one solvent (solvent #1) in which your desired compound is soluble at the boiling point. The second solvent (solvent #2) should induce crystallization when added to a saturated solution of your compound in the primary solvent.

Reference:

See Zubrick pages 114–117.

Recrystallization Steps:

1) The first step is to remove insoluble material from your compound by filtration.

2) Transfer the material to a 50-mL Erlenmeyer flask, equipped with a stir bar. Add an excess amount of solvent #1 (about 20 mL in experiment 3.1) and heat to boiling on a stir/hot plate. The excess solvent is used to keep the compound from precipitating during the filtration.

3) Filter off any insoluble contaminants through fluted filter paper in a pre-warmed stemless funnel (pre-warm by adding some hot solvent just before you filter the solution, thus preventing loss of material on the filter paper.)

4) Wash the flask and filter paper with about 2 mL of hot solvent.

5) Reduce the volume of the solution (to about 15 mL) by boiling off the excess solvent.

6) Cool to room temperature. At this point, it is probably not a saturated solution, so crystallization will not occur.

7) Add solvent #2 dropwise until the solution just becomes cloudy. Again heat the solution to the boiling point (with stirring!) and continue addition of solvent #2. After each drop, you will notice a cloudiness that dissolves away. Continue dropwise addition of solvent #2 until the solution is saturated (i.e. if you were to add one more drop, the cloudiness would

persist, and the solution would be super-saturated.) If this happens, add a drop of solvent #1 to return to a clear solution.

8) Remove the flask from heat, fish out the stir bar with a magnet, allow to cool undisturbed to room temperature before placing in an ice bath.

9) Chill a mixture of the solvent system (in about the same ratio you used to obtain a saturated solution). This will be used to wash your crystals.

10) Collect the crystals on a small Büchner funnel by vacuum filtration, and rinse with the cold solvent mixture.

11) Pull air through the filter cake, then dry thoroughly *in vacuo* before obtaining a yield. One option to dry your product is to place it in a pre-weighed vial, and place the vial in a vacuum desiccator. You can cover the vial by fastening a Kimwipe on top with a rubber band.