

8.6 - 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 105-106, Mohrig 183-197

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 this case) and heat to boiling on a stir/hot plate. The excess solvent is used to keep your compound from precipitating during the filtration.
- 3) Filter off the insoluble contaminant through fluted filter paper in a pre-warmed stemless funnel (pre-warm by adding some hot solvent just before you filter your 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 your solution to about 15 mL by boiling off the excess solvent.
- 6) Cool to room temperature. At this point, you probably do not have 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 the 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 in solution: "super-saturated". If this happens, add a drop of your first 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, then place in an ice bath.

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

10) Collect your 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).

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