

Determination of the Correct Recrystallization Solvent

Heat a beaker of ~100 ml of distilled water to boiling on a hotplate for use as a hot water bath to heat test tubes. Label a set of test tubes with the solvents to be tested: **hexane, water, CH₂Cl₂, acetone and ethanol.**

Grind some acetanilide in a mortar & pestle for use in testing solubility. Place ~100 mg (~1/8") of the powder in each test tube. Add 2 ml (~1") of solvent to each test tube. Shake the test tubes to determine solubility at RT. Record your solubility results at RT.

For the solvents in which the acetanilide did NOT dissolve at RT, immerse the test tube into the hot water bath and check solubility at the BP of the solvent. Shake the hot test tubes to determine solubility. Record your solubility results at BP.

NOTE: hexane has a low BP and will evaporate rapidly in the hot water bath.

The solvent in which the acetanilide did not dissolve at RT, but was soluble at its BP is the recrystallization solvent to use. Record which solvent was determined to be best.

Recrystallization of Acetanilide

Obtain a vial of acetanilide unknown and a vial of activated charcoal (AC). Be sure to record your unknown number. Grind the acetanilide, save a small amount for MP measurement. Transfer the remainder to a piece of tared weighing paper, record the mass and prepare a solution of this in the desired solvent in your 50 ml erlenmeyer flask. (Use 1g/20 ml as the solubility of acetanilide in the boiling solvent to estimate the volume required for your sample.) Record the volume determined. Some material will remain undissolved, as this is the insoluble impurity intentionally added to the acetanilide unknowns. Add 2-3 spatulas of activated carbon and heat the flask on the hotplate.

Obtain a stemless funnel, 250 ml beaker, large watch glass and 9cm filter paper to prepare a hot filtration setup. Add ~1/4' of solvent to the beaker and place the hot filtration setup on the hotplate to heat. Have an additional 50 ml of solvent heating in your 125 ml Erlenmeyer flask to use for washing during the hot filtration step.

When the sample is boiling, pour it rapidly through the hot filtration setup and replace the watch glass cover. When the solvent has passed through the filter paper use additional hot solvent to rinse the 50 ml erlenmeyer flask and rapidly transfer this to the hot filtration setup. Rinse the filter paper with additional hot solvent if any shiny crystals are visible.

When hot filtration and rinsing are complete, remove the funnel and watch glass and allow the solvent in the beaker to evaporate down to the volume originally determined to be needed to dissolve your sample.

When the volume has been reduced adequately, cover the beaker with the watch glass and set it on the desktop to cool. Crystals should form on cooling. When the sample is at ~RT, place it in an ice bath to further reduce its solubility. Also place a few ml of solvent in a small beaker in the ice bath to cool for use in rinsing the crystals during the final vacuum filtration step.

NOTE: If no crystals form on cooling, you may still have too much solvent present. You may also try stirring and scratching the beaker with a glass stir rod to induce crystallization.

Assemble a vacuum filtration setup and pour the cold solution containing your crystals through it. To wash the crystals with cold solvent, disconnect a vacuum hose, add cold solvent, then reconnect the vacuum. Allow the vacuum to run for 5-10 min to dry the crystals. Disconnect the vacuum hose, then shut off the aspirator.

Weigh the collected crystals. Record the mass, calculate % recovery and determine MP range. Also run the MP of the sample of acetanilide prior to recrystallization for comparison. Record the brand and # of the MP apparatus used. Calculate % error for both MPs. Tabulate results, Show sample calculations. Comment on the outcome of the experiment.