

Recrystallization

Recrystallization is a procedure used to purify compounds obtained from natural sources or synthesized in the laboratory. It is a rare reaction that results in no side products or contaminants in the product. Impurities could include soluble, insoluble or coloured by-products.

The steps commonly used to recrystallize a compound are:

1. Carry out solubility tests to determine a suitable solvent
2. Dissolve the solute in a minimum of near-boiling solvent
3. Allow the solution to cool slowly and undisturbed to room temperature then possibly to ice temperature
4. Collect the crystals by filtration
5. Rinse the crystals with a minimum amount of ice-cold solvent
6. Dry the recrystallized product

In CHEM 123L step 1 will be done for you, a suitable solvent for recrystallization will always be given in the laboratory manual. You should know however that the solvent is chosen such that the desired product is soluble in hot solvent and much less soluble in the same solvent at room temperature. The solvent is also chosen so that any suspected impurities are soluble in the solvent at room temperature.

Step 2: Dissolve the solute in a minimum of near-boiling solvent: At this point insoluble contaminants can be filtered out of the solution using a hot filtration technique. You will not use this technique in CHEM 123L, but it is common in some advanced chemistry laboratories.

Step 3: Allow the solution to cool slowly and undisturbed to room temperature: Allowing the solute to form crystals slowly will result in the purest crystals. Fast recrystallization traps impurities within the lattice of the crystal structure. If cooling to room temperature does not produce crystals, the solution can be further cooled in an ice bath. Once crystals start to form, the solution should be removed from ice and allowed to crystallize slowly at room temperature. Crystal formation can also be initiated by scratching the sides of the flask with a glass rod. This introduces tiny glass particles into the solution which act as seed crystals to initiate crystal formation.

Steps 4-6: Collect the crystals using filtration: In CHEM 123L you will use vacuum filtration to isolate your purified crystal product. To aid in transfer of the crystals from flask to funnel use a minimal amount of cold solvent (this prevents dissolution of the newly formed crystals) and use this solvent to rinse the crystals on the filter paper. Dry the crystals by allowing the vacuum to run for approximately 5 minutes, drawing air through the product.