

Recrystallization

What is it, and how do we do it?

The process of recrystallization is used to purify a solid compound. The theory behind it reasons that as crystal X begins growing in solution, molecules of X will fit the crystal lattice better than molecules of Y , and X will be added preferentially. Thus, crystals of pure X will form, and excess Y is left behind in solution.

Recrystallizing from solvent mixture A-B

Great in theory, but how does it work in practise? The easiest way of recrystallizing a compound is when the compound is very soluble in solvent A , and insoluble in solvent B . Thus, one only needs to add solvent B slowly to a solution of the compound X in the minimum amount of solvent A and presto - pure crystals of X come out of solution. There are two problems with this method, however: One, the two solvents A and B must be miscible, which is not always the case and Two, the compound must have the solubility properties described above. This is often not the situation.

Recrystallizing from solvent A

What now? To remedy the problem, and still use our recrystallization technique, we rely on the fact that most substances are more soluble in a hot solvent than a cold solvent. Say for example that compound X is soluble in acetone to the extent of 2 g/100 mL at 0 °C, and 12 g/100 mL at 56 °C (the boiling point of acetone). If we make a saturated solution of X (12 g in 100 mL of acetone) at the boiling point of acetone, and allow the solution to cool to 0 °C, we should then recover 10 g of X . The most noticeable problem here is that sometimes the compound will decompose in the hot solvent - particularly many organometallic compounds.

The steps to follow are:

Dissolve in the minimum amount of hot solvent (rather than heating the solvent + solid).

Filter hot using a gravity filter system.

Cool the filtrate to crystallize.

Isolate by gravity filtration - in batches if necessary.