Purifying a Solid by Recrystallization

Recrystallization is an important technique for purifying solid organic compounds. It is based on the principle that solids are more soluble in hot solvents than in cold solvents. The compound is dissolved in the smallest possible amount of boiling solvent and then the solvent is allowed to cool, and the compound precipitates out. Hopefully, the impurities remain in the cold solvent, and only the pure compound precipitates out. The slower the crystals form, the purer they will be, since fast precipitation may drag impurities into the crystals.

The most important factors in a recrystallization are:

- a) choosing a good solvent
- b) using the right amount of solvent.

A good recrystallization solvent for a particular compound is one that has a polarity which is neither very similar to the compound nor too different from the compound. If the solvent polarity is too similar, the compound will dissolve, but won't come out of solution. If the solvent polarity is too different, the compound won't dissolve at all. If the solvent polarity is moderately different, then temperature will have a big impact on the solubility, allowing the compound to dissolve in boiling solvent but crystallize out when the solvent is cooled.

Getting the right amount of solvent is a matter of proper technique. Too little solvent won't dissolve all of the compound and it won't be purified; too much solvent, and most of the compound will remain dissolved and fail to recrystallize.

Procedure:

- To perform a recrystallization, heat about 10 ml of the recrystallization solvent to boiling in a beaker in your hot plate use a stir bar to keep it from bumping.
- Add the boiling solvent slowly to the solid (in a separate beaker) with a pipet, stirring it with a spatula and heating it on the hot plate until the solid has dissolved and the solvent is still boiling.
- Remove the beaker from the heat and let it slowly cool. It may help to scratch the inside of the beaker with a spatula this gives the crystals somewhere to start forming.
- If no crystals or only a few crystals form, put the beaker back on the hot plate and carefully boil off some of the solvent, stirring constantly, then let it cool again.
- When you have crystals and the beaker is cool enough to handle comfortably, cool it in an ice bath.
- While the solution is still cold, separate the crystals from the liquid by filtration. Allow the crystals to dry, and obtain their mass.