Recrystallization-Purpose

(Palleros, please read Unit 4, pp. 61)

Recrystallization is a very common technique used to purify solids. A material termed *impure* is a mixture of two or more components, from which only one component is desired. The other components are the *impurities*. Thus, recrystallization and other methods of purification are methods of *separation*.

Besides purifications, there are also separations in which all of the components of the mixture are desired and must be separated from each other. Many experiments in this semester will involve a separation method.

Recrystallization-Procedure

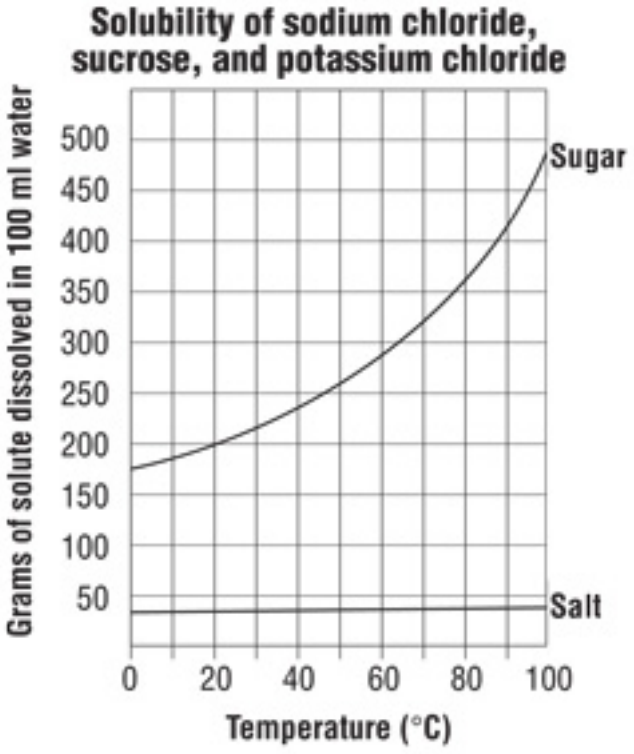
1) Finding a Good Solvent
2) Dissolving the sample
3) Hot filtration
4) Cooling the hot filtrate
5) Separating the crystallized product from mother liquor
6) Washing the filtered product & drying

Checking purity by m.p. test & efficiency by mass recovery (weight, yield)

Recrystallization – The Principles

• Generally, solubility of an organic covalent compound increases as temperature of the solvent increases.

Solubility of sucrose vs KCl in H2O



Selection of Solvent-*Guideline*

- Rule of thumb–"like dissolves like"
- Polar compounds (alcohols, carboxylic acids, amines, amides) are more soluble in polar solvents
- Non polar compounds (hydrocarbons, ketones, aldehydes, ethers) tend to be more soluble in nonpolar solvents

Bottom line

The compound to be purified should be very soluble in the hot solvent but have very limited solubility in the cold solvent.

What makes a good recrystallizing solvent?

A. A solvent that will NOT dissolve the solid while at room temperature

B. The same solvent SHOULD dissolve the solid while at elevated temperatures

C. The cold solvent must keep impurities dissolved in it

D. Usually a good solvent can only be found by trial and error

This week's Recrystallization

Procedure

1) Dissolving the crude sample (impure acetanilide) in water
2) Hot filtration
4) Cooling the hot filtrate to room temp.
5) Cold filtration
6) Washing & drying the product

Checking purity by m.p. test & efficiency by mass recovery (weight, yield)