

Recrystallization is the most important method of purifying nonvolatile organic solids. Recrystallization involves dissolving the material to be purified (the solute) in an appropriate hot solvent. As the solvent cools, the solution becomes saturated with the solute and the solute crystallizes out (reforms a solid). As the crystal develops, impurities are excluded from the crystal lattice, thereby completing the purification process. The crystals can then be collected, washed, and dried. Note: The attractive forces that hold solute molecules to other solute molecules (usually Van der Waals interactions) are overcome during the recrystallization process. Recrystallization does not involve breaking any chemical bonds.

The following is an outline of the recrystallization process.

1. Pick the solvent. In the introductory organic lab course, the solvent for recrystallization is usually determined for you. The criteria used to choose an appropriate recrystallization solvent includes:

- a. finding a solvent with a high temperature coefficient. The solvent must not dissolve the compound at low temperatures (that includes room temperature), but must dissolve the compound at high temperatures. The solute must dissolve in order to rid its lattice of impurities, but must not remain dissolved at room temperature (after all, recovery of the solid is essential!).
- b. using a solvent that dissolves impurities readily or not at all. If the solvent dissolves the impurities readily (even at room temperature) then the impurities will not become trapped in the developing crystal lattice, but will remain dissolved in the solvent. If the impurities do not dissolve (even at elevated temperatures) then they can be easily

removed by gravity filtration. (see the section on gravity filtration)

- c. insuring the solvent will not react with the solute. As mentioned earlier, recrystallization does not chemically alter a molecule. No chemical bonds must be broken in the solute molecule. The crystal lattice is dissolved at elevated temperatures, but this only involves overcoming the intermolecular attractive forces.
- d. using a solvent that is nonflammable, inexpensive and volatile. Solvents with low boiling points (i.e., volatile) can be easily removed from the resultant crystals by simply allowing the solvent to evaporate.

2.Dissolve the solute. Remember that the solute should dissolve only when the solvent is heated. Therefore, the solvent is heated to its boiling point (remember to use boiling stones!) and then slowly added to completely dissolve the solute. If too much solvent is added, the solution will not be saturated upon cooling and no crystals will form.

Dissolving the solute generally involves adding a small volume of hot solvent, swirling the flask (or stirring the solution), and watching to see if the solute dissolves.

3.Decolorize the solution. If the solute is supposed to be white in its pure solid state (most organic solids are) and the solution is colored after dissolving all the solute, it will be necessary to add decolorizing carbon to the solution. This will cause the colored molecules to adsorb onto the surface of the decolorizing carbon, thereby ridding the solution of these impurities. Should

these impurities remain in solution, they may become trapped in the developing crystal during cooling. Review the material about decolorizing carbon.

4.Filter any solids from the hot solution. If decolorizing carbon was used (as in step 3) or undissolved impurities remain in the hot solution, it is necessary to gravity filter the solution while it is still hot. Review the information about hot gravity filtration and decolorizing carbon. Under no circumstances should the hot solution be vacuum filtered with a Buchner funnel. This leads to premature crystal development as the solution passes through the vacuum filter. (The vacuum reduces the pressure, but also the temperature.) Impurities will be trapped in the crystal lattice and steps 1 through 3 will need repeated!

5.Crystallize the solute. This involves allowing the hot solution with the solute dissolved to return to room temperature slowly. The slower the cooling process, the less chance of trapping impurities in the developing crystal lattice. Allow the solution to reach room temperature. If crystals have not formed by the time the solution reaches room temperature, further steps may be necessary to induce nucleation. Try the following:

- a. Take a clean, glass rod and scratch the inside surface of the Erlenmeyer flask. This provides a small speck of glass upon which nucleation may occur.
- b. Ask a classmate who has recovered the pure solute to 'donate' a small amount of the solute. Add a small sample of the pure solute to the flask. Again, this is thought to provide a site for nucleation.
- c. Place the Erlenmeyer flask in an ice water bath. This will dramatically reduce the temperature of the solution. Saturation conditions may be met at this lower temperature, allowing the formation of crystals.

5. Collect and wash the crystals. The resultant crystals formed via this process can be collected by vacuum filtration, provided the solution is at room temperature and no further crystal growth is evident. To transfer all of the crystals to the Buchner funnel, add a small amount of the cold recrystallization solvent. Remember, the solute is not soluble in the cold solvent, so it is safe to use this solvent to transfer the crystals. Wash the crystals with a small quantity of the cold solvent to rinse any impurities off the crystal surface.

6. Dry the crystals. Usually a melting point is taken directly following the purification process, so it is necessary to quickly dry the crystals. This is accomplished by allowing the crystals to remain in the Buchner funnel and keeping the vacuum on for a few minutes. Alternatively, if more time is allotted, the crystals can be stored safely and allowed to sit for a few days, allowing the solvent to evaporate over time.