# Recrystallization

When trying to isolate a compound from a mixture, such as caffeine from tea or coffee, many steps are usually required to obtain a pure sample. As discussed previously, one method often used is a liquid/liquid extraction. In the previous experiment, three impure unknown solids were isolated from an initial one-gram sample. These solids, often referred as crude, have impurities present usually in small amounts. An old but very effective method to separate out these impurities is called recrystallization. In the teaching laboratory a recrystallization usually involves adding the crude solid to a flask containing a solvent. The solvent is heated to its boiling point to dissolve the solid. Finally, the solution is slowly cooled to induce the precipitation of the solid which can then be isolated. Although a recrystallization is a relatively straight forward process, to achieve optimal results requires great care. The entire process involves several steps, which are listed below.

**I.** Select the correct solvent.

**II.** Dissolve the solid in the solvent at an elevated temperature. Impurities may need to be removed at this point by either filtration or addition of activated charcoal.

**III.** Cool the solution to induce crystallization of the compound.

**IV.** The crystals are isolated by filtration and then dried for analysis.

Selection of the solvent is the most important step when starting a recrystallization. The solvent must fulfill a set of criteria for it to be considered. The first criteria the solvent must meet is that the compound be insoluble, or nearly insoluble, in “cold” solvent. In most instances the term cold refers to room temperature. The second criteria for the solvent is that the compound be soluble, or mostly soluble, in “hot” solvent, which usually means boiling or near boiling. Finally, the last criterial is that the solubility of the impurities in the solvent be temperature independent. In other words, the impurities are either completely soluble or completely insoluble regardless if the solvent is hot or cold.

The identification of the correct solvent can be a lengthy process usually requiring trial and error. The polarity of the compound should be like the solvent chosen. Polar compounds usually dissolve in polar solvents and non-polar compounds dissolve in non-polar solvents. Another important factor to be considered is the boiling point of the solvent. In general, it is a good idea for the solvent to have a boiling point below the melting point of compound being recrystallized. The solvent should also have a high enough vapor pressure to enable faster drying of the crystals once they have been isolated. Finally, the solvent should not react with the compound being purified.

Although it can be an arduous task to identify the recrystallization solvent, for most undergraduate laboratory experiments the solvent has already been selected. Once the solvent has been selected, the next step of a recrystallization is to dissolve the compound. The solid is placed in an appropriately sized Erlenmeyer flask and with a small amount of solvent; the amount added is not enough to dissolve it when hot. The flask is heated to bring the solution to a

gentle boil. Once boiling, solvent is added in small portions until the compound just dissolves. It is important to keep to solvent volume to a minimum to maximize yield. The additional solvent being added should ideally be hot to ensure adding a minimal amount. If this is not possible and addition of the solvent causes the solution to cool, ensure that the solution is returned to boiling before the next portion is added.

After most of the sample has dissolved and additional solvent does not dissolve the remaining solid, the remaining solid impurities need to be removed. Removal of the impurities usually only requires vacuum filtration using a Buchner funnel. The filtration system needs to be setup and ready for use so that the solution can be filtered while still hot. Sometimes the impurities are colored and cannot be removed by filtration remaining even after recrystallization. If this occurs, addition of a small amount of activated carbon will often absorb the impurity. The carbon can then be filtered which also removes the impurity.

After filtration, or if filtration is not needed, the solution needs to be cooled to room temperature. Cool the solution slowly to ensure the formation of good crystals. While cooling, the flask should be covered to prevent anything from the air contaminating the solution. Do not agitate the flask while it is cooling, or the crystals that form might be too small. If the solution has reached room temperature and crystals have formed, there are a few tricks to induce crystallization. Crystals can be induced to form by cooling it further in and ice-water bath, seeding it, or scratching the side of the flask with a glass rod. Once crystals have formed, they can be isolated by vacuum filtration. Once isolated, the crystals must be dried before analysis. The crystals can be dried by placing them in a watch glass, a beaker, a vial or some other container to allow them to air dry.

## References


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