The principle behind recrystallization is that the amount of solute that can be dissolved by a solvent increases with temperature. In recrystallization, a solution is created by dissolving a solute in a solvent at or near its boiling point. At this high temperature, the solute has a greatly increased solubility in the solvent, so a much smaller quantity of hot solvent is needed than when the solvent is at room temperature. When the solution is later cooled, after filtering out insoluble impurities, the amount of solute that remains dissolved drops precipitously. At the cooler temperature, the solution is saturated at a much lower concentration of solute. The solute that can no longer be held in solution forms purified crystals of solute, which can later be collected.

Recrystallization works only when the proper solvent is used. The solute must be relatively insoluble in the solvent at room temperature but much more soluble in the solvent at higher temperature. At the same time, impurities that are present must either be soluble in the solvent at room temperature or insoluble in the solvent at a high temperature. For example, if you wanted to purify a sample of Compound X which is contaminated by a small amount of Compound Y, an appropriate solvent would be one in which all of Compound Y dissolved at room temperature because the impurities will stay in solution and pass through filter paper, leaving only pure crystals behind. Also appropriate would be a solvent in which the impurities are insoluble at a high temperature

because they will remain solid in the boiling solvent and can then be filtered out. When dealing with unknowns, you will need to test which solvent will work best for you. According to the adage "Like dissolves like," a solvent that has a similar polarity to the solute being dissolved will usually dissolve the substance very well. In general, a very polar solute will easily be dissolved in a polar solvent and will be fairly insoluble in a non-polar solvent. Frequently, having a solvent with slightly different polarity characteristics than the solute is best because if the polarity of the two is too closely matched, the solute will likely be at least partially dissolved at room temperature.

There are five major steps in the recrystallization process: dissolving the solute in the solvent, performing a gravity filtration, if necessary, obtaining crystals of the solute, collecting the solute crystals by vacuum filtration, and, finally, drying the resulting crystals.

1. **Dissolving the solute in the solvent**
   a. Add a small portion of boiling solvent to the beaker that contains the impure sample and a boiling chip.
   b. Heat the beaker containing the solute and continue adding boiling solvent incrementally until all of the solute has been dissolved. If additional solvent can be added with no appreciable change in the amount of solute present, the particulate matter is probably insoluble impurities.

2. **Hot Gravity Filtration**
   a. This step is optional if there is no visible particulate matter and the solution is the expected color (most organic compounds are white or light yellow)
   b. If the solution is not the expected color, remove the boiling solution from the heat and allow it to cool to beneath the boiling point of the solvent. Add a small amount of activated carbon (about the size of a pea) and mix the solution. If too much activated carbon is used, excessive loss of the desired product will result. Boil the solution containing the activated carbon for 5 to 10 minutes. A filter aid will need to be placed in the filter paper to remove the carbon in the following steps.
   c. Flute a piece of filter paper and place it inside of a stemless funnel. A funnel with a stem is prone to premature recrystallization inside the stem because the filtrate can cool as it passes through the stem. At these cooler temperatures, crystals are likely to form.

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   d. Heat a beaker that contains some of your recrystallization solvent. Place the funnel and filter paper assembly in the beaker so that the rising vapors from the boiling solvent can heat the funnel and filter paper. Having the set up heated

before filtration will prevent crystals from forming on the paper and in the funnel (see Figure 1 below).

Figure 1. Hot gravity filtration. Keeping the set up hot prevents crystals from forming prematurely.

e. Keeping the solution very hot so the solute stays dissolved, pour the solution through the funnel and filter paper assembly. As the filtrate begins to accumulate, heat the receptacle beaker; the resulting vapors will help to prevent any crystallization in the funnel or on the filter paper.
f. If the funnel was properly heated before filtration, all of the solution will have passed through and no crystals will have formed on the paper or in the funnel. If crystals have formed, pouring a small amount of boiling solvent through the funnel will dissolve these. If the solution is still discolored after using activated carbon and filtering, either the color is from the compound and will not go away or you need to repeat the step with the addition of activated carbon.

g. The solution should be allowed to cool slowly to room temperature. Gradual cooling is conducive to the formation of large, well-defined crystals.

3. **Vacuum Filtration**

(see Filtering Techniques, remembering these additional points)

a. Agitate the crystals with a fire polished glass-stirring rod before pouring the mother-liquor along with the crystals through the Buchner funnel. Apply the maximum amount of suction possible using the aspirator.

b. Some crystals may have been left behind in the beaker; there are two ways to effect a quantitative transfer of all of this material. Either use a portion of the filtrate to rinse the beaker or use a rubber policeman on the end of your stirring rod to scrape the remaining crystals into the Buchner funnel.

c. When the crystals have been collected and washed, allow the aspirator to run for several minutes so that the crystals have an opportunity to dry.

4. **Drying the Crystals**

a. When the crystals have been dried as much as possible in the Buchner funnel, use a scoopula to remove them to a beaker or crystallizing dish. This will ensure that the crystals are not contaminated by filter paper fibers as they dry.

b. After removing all the crystals from the filter paper, remove the filter paper and scrape any remaining crystals from the

funnel.

c. Spreading the crystals out in a beaker or a crystallizing dish will provide for the most efficient drying as the crystals will have a maximum of exposed surface area.

d. When the crystals are dried, the purity of the sample can be measured by performing a melting point determination.

5. **What to do if crystals don't form**

If crystals don't form upon slow cooling of the solution to room temperature there are a variety of procedures you can perform to stimulate their growth. First, the solution should be cooled in an ice bath. Slow cooling of the solution leads to slow formation of crystals and the slower crystals form, the more pure they are. Rate of crystallization slows as temperature decreases so cooling with an ice bath should only be used until crystals begin to form; after they do, the solution should be allowed to warm to room temperature so crystal formation occurs more slowly. If no crystals form even after the solution has been cooled in an ice bath, take a fire polished stirring rod and etch (scratch) the glass of your beaker. The small pieces of glass that are etched off of the beaker serve as nuclei for crystal formation. If crystals still do not form, take a small amount of your solution and spread it on a watch glass. After the solvent evaporates, the crystals that are left behind can serve as seeds for further crystallization. Both these methods of nucleation (i.e. etching and seed crystals)

cause very rapid crystallization, which can lead to the formation of impure crystals.

Crystals will not form if there is a large excess of solvent. If no crystals form with the methods already discussed, a portion of the solvent may need to be removed. This can be accomplished by heating the solution for a period of time in order to evaporate some solvent. The new, concentrated solution, should be cooled, and the previously mentioned methods to stimulate crystallization should again be attempted.

Another potential problem in recrystallization is that the solute sometimes comes out of solution in the form of an impure oil instead of forming purified crystals. This usually happens when the boiling point of the solvent is higher than the melting point of the compound, but this is not the only scenario in which this problem presents itself. If this begins to happen, cooling the solution will not stimulate crystallization, it will make the problem worse. If an oil begins to form, heat the solution until the oil portion dissolves and let the whole solution cool. As the oil begins to form again, stir the solution vigorously to break up the oil. The tiny beads of oil that result from this shaking may act as the nuclei for new crystal formation.

NEXT: Selecting a Solvent