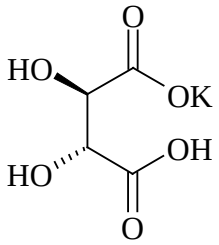
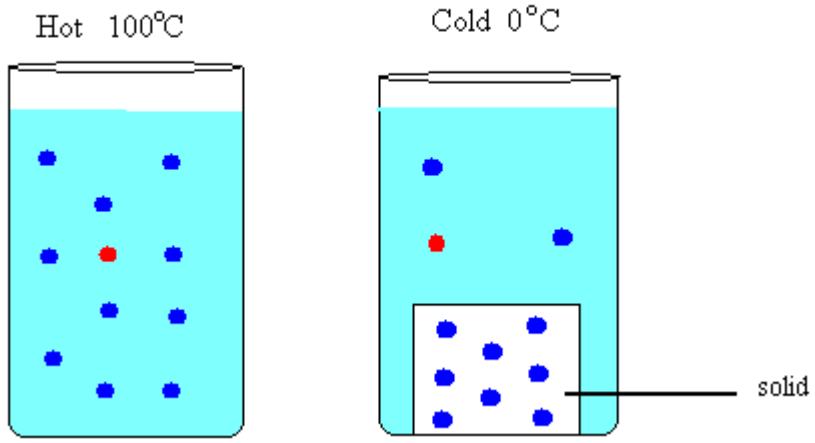
Recrystallization



Cream of Tartar

To recrystallize a substance, one dissolves the solid in a minimum amount of a hot solvent. The mixture is allowed to cool and because generally, most compounds are more soluble at higher temperatures, as the mixture cools, the solubility decreases and some of the solute crystallizes. For maximum percent recovery, it is important to choose a solvent in which the solute has a high solubility at high temperatures and a low solubility at low temperatures.

How does this purify the compound? Lets assume that the solute has a solubility in the solvent at 0°C of 2.00 grams of solute per 100 ml of solvent and 10 g of solute per 100 ml at 100 °C. In the picture below, each gram of solute is represented by a blue dot. Lets assume that an impurity is also present and it has the same solubilities. Fortunately, only 1 g of the impurity is present. The impurity is represented by a red dot. You can see that the impurity will not crystallize out while 8 grams of the pure solute can be isolated. Notice that some of the solute is lost. Of the original 10 grams of solute, only 8 grams are isolated, 2 grams are lost to the solution (called the mother liquors).



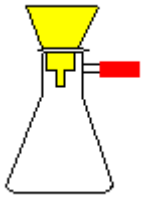
A crystal is a regular arrangement of solute particles. Because the impurity has a different shape, it will tend to be excluded from the crystal.

Experiment

You will purify a store sample of "Cream of Tartar" by recrystallization from water.

1. Obtain and weigh a sample of potassium hydrogen tartrate; use between 200 and 300 mg.
2. In a boiling water bath, heat a sample of distilled water.

3. Dissolve your sample in a minimum of water. To do this, add the water drop-wise to your solid, stirring well between each drop, until the solid just dissolves. You might have to heat the test tube while you are adding the water to keep the solution hot.
4. After getting the solid to dissolve, allow the solution to cool slowly and look for crystals. After the solution has cooled to close to room temperature, cool it in an ice bath. Collect the solid using vacuum filtration and your Hirsh funnel.
5. Weigh the solid and after drying, find the melting point using the Mel-Temp apparatus.



Calculations:
Determine the % Recovery:

$$\%\text{recovery} = \frac{\text{amount recovered}}{\text{initial amount}} x100\%$$

Post Lab Questions

1) Define the following terms:
   a) saturated solution
   b) supersaturated solution
   c) solubility
   d) mother liquor
   e) precipitate
2) Why is the recrystallized substance washed with solvent while on the filter?
   a) Why does the solvent used in this step need to be ice-cold?
3) What prevents soluble impurities from appearing in the final product during recrystallization?
4) Suppose not all the solvent was removed from a recrystallized solid. What would the effect be on the melting point of the solid?
5) Name two ways of inducing crystallization.

Reading
General
http://chemlab.pc.maricopa.edu/labbooks/230/recrystallization/index.html
Why slow cooling?
http://orgchem.colorado.edu/hndbksupport/cryst/crystdiag.html
Inducing crystallization.
http://www.chem.wisc.edu/areas/organic/orglab/tech/induce.htm