Recrystallization

Recrystallization technique

Recrystallization is a laboratory technique used to purify solids based on their different solubilities.

A small amount of solvent is added to a flask containing an impure solid. The contents of the flask are heated until the solid dissolves.

Next, the solution is cooled.
A more pure solid precipitates, leaving impurities dissolved in the solvent.

Vacuum filtration is used to isolate the crystals.

The waste solution is discarded. The impurity either remains dissolved or removed before cooling by absorption on decolirizing carbon (Norit)

Selection of solvent: characteristics

An ideal crystallization solvent should have:

a) It should dissolve a large amount of the substance at its boiling point and only a small amount at room temperature.

b) It should dissolve the impurity at low temperature or not at all.

c) Its boiling point should be lower than the melting point of the solid substance.

d) Upon cooling, the solvent should yield well formed crystals.

e) It should not react with the solute.

f) It should be safe (nonflammable, nontoxic)

g) It should be inexpensive

Selecting a solvent:

Carry out solubility tests on the compound to be re-crystallized.

Place 0.1 g of the powdered solid into a clean test tube. Add the solvent dropwise with shaking.

If the solid dissolves, the solvent is not fit for crystallization.

If it does not dissolve, warm the solution gently to boiling. If it partially dissolves, add more solvent and warm.

Cool the solution and observe the appearance of crystals.

Note: if the solid does not dissolve in a large amount of the hot solvent, then it is not fit for re-crystallization.

Summary of Recrystallization Steps

The **objective** is to dissolve the solute in a minimum amount of the hot solvent.

1. Select the proper solvent (see the notes below)
2. Place a small amount of solid in an Erlenmeyer flask. Add a boiling chip.
3. Add a small quantity of the appropriate hot solvent .
4. Heat to boiling on a steam bath (hot plate or Bunsen burner can be used).
5. To the boiling solution, add more hot solvent in small portions with stirring.
6. Continue addition of solvent until all solute dissolves at boiling.
7. If colored impurities are present, add a small amount of charcoal to the solution (not boiling solution) and boil.
8. Filter the hot solution by gravity using a preheated short stem funnel. Apply heat to dissolve the solid.
9. Cool the solution to crystallize the product. Slower cooling may lead to a higher purity product, so it's common practice to allow the solution to cool to room temperature before setting the flask in an ice bath.

Summary of Recrystallization Steps-Continued:

10.Crystals usually begin forming on the bottom of the flask.

* It is possible to aid crystallization by scratching the flask with a glass rod at the air-solvent junction (assuming you are willing to purposely scratch your glassware).

The scratch increases the glass surface area, providing a roughened surface on which the solid can crystallize.

- Another technique is to 'seed' the solution by adding a small crystal of the desired pure solid to the cooled solution. Be sure the solution is cool, or else the crystal could dissolve.
- If no crystals fall out of solution, it's possible too much solvent was used. Allow some of the solvent to evaporate. If crystals do not spontaneously form, reheat/cool the solution.

11.Use vacuum filtration to collect the pure crystals

12- Wash the crystals with a small amount of the cold solvent and press out the solvent.

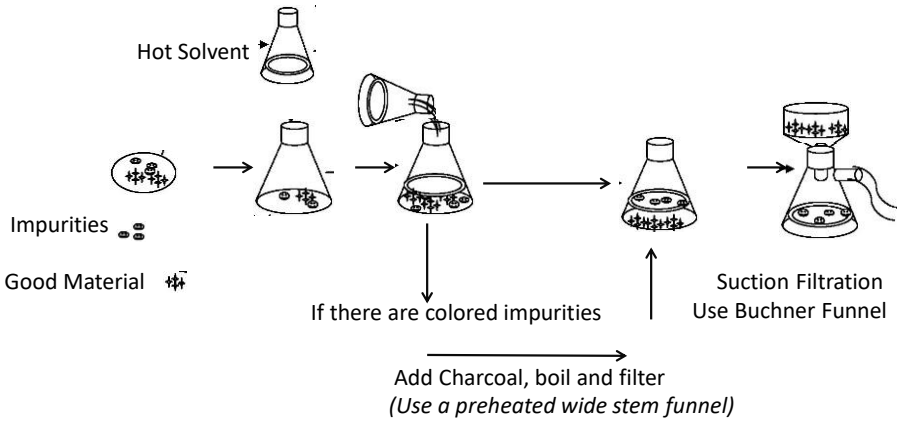
13.The crystals can be air dried or placed in a vacuum desiccators.

Decolorizing:

If there are colored impurities, the solution (in step 3 above) is cooled.
Charcoal (2-3%) of the weight of sample is added to the cold solution which is then boiled with stirring and filtered. (Steps 4 & 5 above).

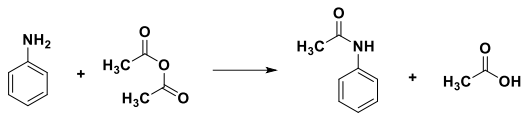
Summary of steps

1)Transfer the solid 2) Heat Solvent 3)Pour hot solvent gradually 4)Pure solid precipitates Impurities remain in solution **5)Filter**



Preparation and Re-crystallization of Acetanilide:

Acetanilide is a white solid that can be prepared by reacting aniline and acetic anhydride:



Aniline + Acetic Anhydride Acetanilide + Acetic Acid

Basic Information:

Compound	Molecular Mass	M.P. /(B.P.)	Density
Acetanilide	135.17	114 ºC	--------
Aniline	93.13	(184 ºC)	1.022 g/mL
Acetic Anhydride	102.09	(138 ºC)	1.082 g/mL

Experimental Procedure:

1- Weigh 3 grams of crude acetanilide in a 250 mL-Erlenmeyer flask.

2- Add boiling water gradually to dissolve all the white solid.

3- Cool a bit and add 0.2 g of decolorizing carbon (Charcoal).

4- Boil the solution again, filter out charcoal using a preheated Buchner funnel and suction flask. Then while hot, transfer the filtrate to clean beaker.

5- Cool the filtrate to room temperature then in ice (crystals should appear).

6- Use suction filtration to collect the crystals on a Buchner funnel. Wash the crystals with a small amount of cold water.

7- Keep the suction on to squeeze out all the solvent. Air dry the crystals.

8- Weigh the dry product, calculate the percentage yield and determine its melting point.

9- Collect the product in a paper and write your name and submit it to your instructor.