Ministry of Higher Education and Scientific Research
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Experimental No.2
Recrystallizing process

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- The Purpose of experiment:

a) To purify samples of organic compounds that are solids at room temperature
b) To dissociate the impure sample in the minimum amount of an appropriate hot solvent

- Introduction And Theory

Recrystallization is the primary method for purifying solid organic compounds. Compounds obtained from natural sources or from reaction mixtures almost always contain impurities. The impurities may include some combination of insoluble, soluble, and colored impurities. To obtain a pure compound these impurities must be removed.

Recrystallization involves dissolving a solid in a solvent and crystallizing it again, taking the opportunity to discard impurities along the way. One normally chooses a solvent in which the solubility increases significantly with temperature. The solid is dissolved in a minimal amount of hot solvent, and the solution is filtered to remove *insoluble* impurities. Upon cooling. the solution, the desired compound crystallizes, leaving *soluble* impurities in solution. Alternatively, a mixed solvent system can be used to modify the solubility — the key is to get the compound into solution then get it back out of solution.

Ideally, one would like to recover all of the desired solid completely free of contaminants. Unfortunately, this is rarely possible. Usually, if most of the material is recovered, it is not very pure, and extremely pure material can be obtained only in low yield. The trick is to find the proper balance between yield and purity.

Now to the specifics of the problem at hand. Over the summer, certain stockroom personnel had an unauthorized juggling contest and accidentally dumped all the acetanilide on the floor. Unfortunately, the floor wasn't very clean, now the material is contaminated with what appears to be rodent hair, gravel, dead spiders, etc. They were able to remove most of the dead roaches. Your mission, should you decide to accept it... well, you really have no choice but to accept it, do you?... is to clean up the acetanilide by recrystallizing it.

Part A — acetanilide. Fetch about 1 g of dirty acetanilide. Don't fool around adjusting the mass to 1.000g — it's not that important — just record **in your notebook** exactly how much you used. Place your weighed sample in a 125-ml Erlenmeyer flask and add a boiling chip. Heat about 50 ml of water to boiling in a separate flask (+ boiling chip!) using a hot plate.

Note: Always add a boiling chip before heating any solvent or solution. Never add any solid to a liquid that is at or near its boiling point. Suddenly creating nucleation sites is likely to cause anything from a simple boil-over to something resembling a volcano.

Now, dissolve the solid in a minimal amount of boiling water. This is done by adding the solvent to the solute (not the other way around!) in small increments and bringing the solution to a boil. Start out with about 10 - 15 ml of water and add a few ml at a time until all the soluble stuff dissolves. (Don't measure it out, just estimate!) Watch how much solid dissolves each time — this will give you an idea how much water you'll need to add to dissolve it all. Just be careful not to add way too much water, or you'll have to reduce the volume later. And keep in mind that there may be insoluble junk remaining after all the good stuff has dissolved. How do you tell the difference? One bit of information is the color. What color is acetanilide? What color is the stuff that's not dissolving? One final bit of advice — solids with melting points below the boiling point of the solvent will melt and form what look like oil droplets. If you see this, you need to add more solvent until the droplets dissolve. Even solids that melt above the solvent bp may do this if they are very impure (remember mp depression?) or if the hot water can infiltrate the crystal lattice.

To get rid of the insoluble bits, filter the hot solution by gravity through a pre-warmed funnel containing fluted filter paper. The tricky part is to keep the solution hot during this operation. This is important — if you heat the solution, then remove it from the hot plate and try to do the filtration on the bench top, everything's going to cool down, right? Solid will precipitate all over the place and make a terrible mess. Rinse the filter paper with a little hot solvent to dissolve any crystals that may have formed.
Slow crystallization is the key to getting high purity product. Plunging the hot solution into ice may cause tiny crystallites to crash out of solution (if it doesn't break the flask), and lots of impurities will end up trapped in the crystal lattice. So don't do that. Instead, allow the filtrate to cool slowly. Be patient. If the solution was close to the saturation point when it was hot, the compound should be eager to crystallize as the solution cools. If no crystals form, this could be because (1) the solution is too dilute (i.e. the solid is completely soluble so it doesn't want to come out of solution), or (2) the solution is supersaturated (i.e. the solid would like to crystallize but can't quite decide how to get started. If the problem is too much solvent, the only remedy is to boil off the excess. If the solution is supersaturated, try scratching the flask with a glass stirring rod — the scratches and glass chips may provide
nucleation sites that get crystal formation started. If that doesn't work, try adding a tiny seed crystal. The best seed crystal is a tiny speck of someone else's clean solid. If you're not too fussy about product purity, those white flakes in your instructor's beard can probably also be used as seed crystals. Once crystal formation appears to have stopped, cool the flask in ice to complete the crystallization. Collect the crystals by suction filtration using a Büchner funnel. Wash the crystals with a few milliliters of *cold* water, and press them down with a clean spatula or small beaker.

Dry the crystals by pressing them between two pieces of clean filter paper, and let them dry until your next lab period. You will then determine the mass, the % recovery, and the melting range.

Part B — solids from Extraction lab. You've saved samples of benzoic acid, benzocaine, and fluorenone that we separated by extraction several weeks ago. Now we're going to purify these by recrystallization. So that we have plenty of material to work with we're going to start by consolidating samples with other groups.

Get together with two other groups, and combine your three samples of benzoic acid, (separately!) combine your three samples of benzocaine, and combine your three samples of fluorenone. (If you misread this and mix everything together, no worries, you can just repeat the extraction to separate them again.)

Now, one group is going to recrystallize the benzoic acid from water, one group is going to recrystallize the benzocaine from ethanol and water, and one group is going to recrystallize the fluorenone from hexanes.

- ## Tools used in the experiment

1. Cups
2. Jugs
3. Cones
4. Volumetric flasks
5. Nomination papers
6. Hot disk
7. Balance

- ## The materials used in the experiment

1. Benzoic acid
2. Distilled water

- The six steps used here to recrystallize a compound are.

1- carry out solubility tests to determine a suitable solvent.
2- dissolve the solute in a minimum of near boiling solvent.
3- allow the solution to cool slowly and undisturbed to room temperature (RT) then possibly to ice temperature.
4- collect the crystals by filtration.
5- rinse the crystals with a minimum amount of ice-cold solvent.
6- allow the crystals to dry.

- Chooses a solvent for Recrystallization

The proper choice of a solvent is an important part of the art of crystallization.
The ideal solvent should.
1- Be chemically inert toward the solute.

2- Dissolve the solute readily at its boiling point but sparingly at low temperature (0 – 25 ^{0}C).

3- Dissolve impurities either very easily or not at all.

4- Not be flammable of low cost and of low toxicity.

Practically to choose a good solvent take about **0.1gm** of the compound to be purified (a pure sample) and try to dissolve it in **1ml** of the solvent if it dissolves in the cold solvent the solvent will not be good for recrystallization if it dissolves in the solvent with heating, the solvent will be good for recrystallization. If it does not dissolve in the solvent even with heating, the solvent will no; be good for recrystallization. Solvent extensively used for recrystallization include water, ethanol, chloroform, ether, acetone, and benzene.

- Procedure (crystallization of benzoic acid):

1- Heat some solvent (water) to boiling .Place the solid (benzoic acid) to be recrystallized in an Conical flask.
2- Pour a small amount of the hot solvent (water) into the flask containing the solid (benzoic acid).
3- Swirl the flask to dissolve the benzoic acid.
4- Place the flask on the steam bath to keep the solution warm.
5-If the benzoic acid is still not dissolved, add a tiny amount more water and swirl again.
6-After a while, crystals should appear in the flask.
7-You can now place the flask in an ice bath to finish the crystallization process.
8-You are now ready to filter the solution to isolate the crystals. Remove the filter paper from the Buchner funnel when done.
9- After your crystals are filtered from the solution, put them on a watch glass.

10- Let the crystal finish drying on the watch glass

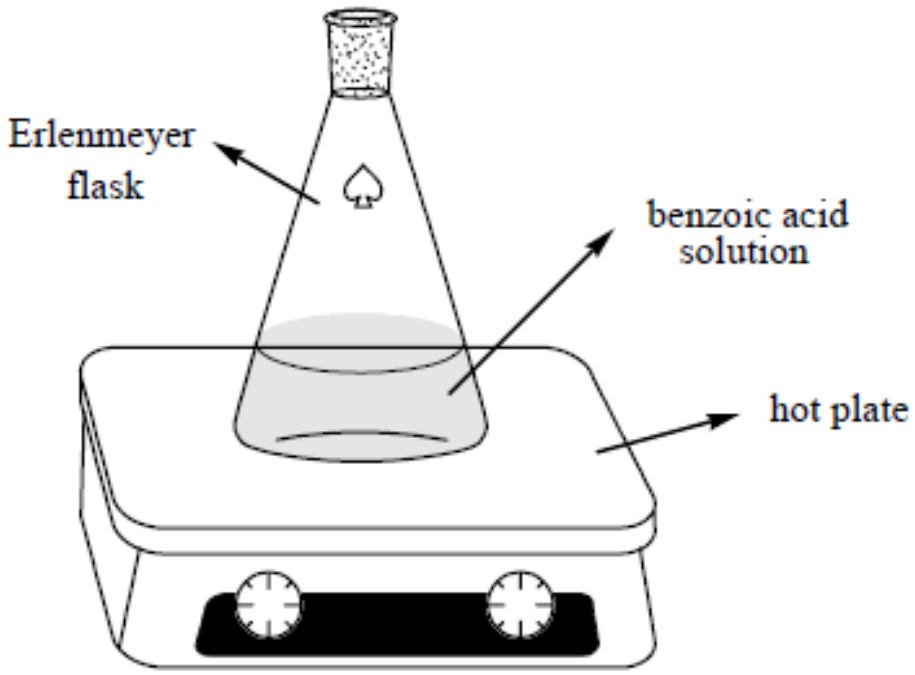

Fig 1- Dissolving benzoic acid

Discussion :

1- What is the The Purpose of experiment?

2- What is the process of re-crystallization ?

3- How is solvent used in the selection process ?

4- What is the sample that used in this experiment ?