



**Ministry of higher Education &  
Scientific Research  
Al-Rasheed University College/  
Pharmacy Department**



**Practical organic pharmaceutical chemistry I  
4<sup>th</sup> stage/ 2<sup>nd</sup> semester  
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**Lab 2  
Recrystallization**

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## Recrystallization

Is the primary method for purifying solid organic compounds. Compounds obtained from natural sources or from reaction, mixtures usually contain impurities. To obtain a pure compound these impurities must be removed.

- The method of recrystallization simply involves the preparation of a saturated solution of the compound to be purified in a small volume of the desired solvent (enough to dissolve the compound) at elevated temperatures (at or near the boiling point of the solvent).
- Charcoal may be used when the compound is contaminated with colored impurities.
- Filtration of the hot solution is then done to separate the insoluble materials or impurities. The hot solution will then be allowed to cool up so that the dissolved compound will crystallize out. After that, the crystals are separated from the remaining supernatant liquid by means of filtrations.
- This collected crystalline solid compound is dried and then tested for purity usually by melting point determination, spectroscopic method or TLC.

### 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 °C).
3. Dissolve impurities either very easily or not at all.
4. Not be flammable of low cost and of low toxicity.
5. Polar compound (polar solvent)-----non polar compound (non-polar solvent)

### ***Recrystallization using mixed solvents***

- The two solvents must be completely miscible with each other such as alcohol and water, ether and pentane, glacial acetic acid and water.
- So the compound is dissolved in the solvent, charcoal is used if required, and the solution is filtered to get rid of the insoluble impurities.
- Then the other solvent (in which the compound is insoluble) is added to the filtrate gradually until turbidity

### ***Notes:***

- The funnel, filter paper and the container of the solution should be kept hot throughout the filtration process to prevent the deposition of the crystals on the filter paper or on the neck of funnel. Therefore, it is recommended to wash the filter paper after completing the filtration process with a small amount of the hot solvent.
- Use a minimum volume of the solvent to prevent the loss of the compound because large volume of the solvent will keep most of the compound dissolve in it.
- Drying of the purified substance can be achieved by air-drying, oven drying, and freeze drying or using a desiccator containing a drying agent as anhydrous calcium chloride.

### **Procedure (recrystallization of benzoic acid):**

- 1- Heat some water to boiling; Place the benzoic acid to be recrystallized in a conical flask.
- 2- Pour a small amount of the hot water into the flask containing the 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- Filter the hot solution 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.