Recrystallization

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Expermental Theory

Recrystallization is one of the most widely used methods for purifying solid organic materials. It involves selecting the crystal of a compound from a mixture of solid organic materials.

Experimental Principle:

It is based on the difference in solubility between compounds. The unwanted substance (or impurities present in the mixture) must have different solubility properties than the desired substance or the substance to be recrystallized in the chosen solvent.

The Steps of the Recrystallization Process Include:

1. Selecting an appropriate solvent.
2. Dissolving the sample in the smallest possible volume of solvent by heating.
3. Decolorizing the solution using activated carbon.
4. Hot filtration.
5. Cooling the filtrate.
6. Filtering under low-pressure vacuum using a Buechner apparatus.
7. Washing the crystals.
8. Drying the crystals.

1- Selecting an Appropriate Solvent

If we take any chemical source, it may help us extract or identify the appropriate solvent for a known or familiar substance. For example, benzoic acid has water as its best solvent.

However, if the compound is unknown, there are no theoretical considerations used in selecting the solvent except one rule, which is “like dissolves like.” Therefore, the suitable solvent must be determined practically (experimentally) depending on the properties of the selected solvent.

The Properties That a Solvent Must Possess:

1. The solvent must be chemically inert toward the solute.
2. It must not dissolve the substance when cold and must dissolve it upon heating (boiling).
3. Impurities must completely dissolve when cold or remain undissolved even when hot (boiling).
4. It must be highly volatile and thus easily evaporate from the crystals.
5. It must be inexpensive, non-flammable, and non-toxic.

2- Dissolving the Sample in the Smallest Possible Volume of Solvent by Heating

Solubility: The disappearance of solute particles between solvent particles.

The dissolution process requires energy. This energy comes from breaking the bonding forces between the particles, where energy is released and utilized in the dissolution process.

3- Decolorizing the Solution Using Activated Carbon

That means removing the color caused by impurities through the adsorption and absorption of impurities onto the surface of activated carbon particles.

4- Hot Filtration

A- To remove insoluble impurities and activated carbon.

B- To prevent precipitation of the desired material (to prevent the loss of even a small amount of the desired compound).

5- Cooling the Filtrate

There are two types of cooling:

A. Rapid cooling, which produces many small crystals.

B. Slow cooling, which produces few large crystals.

Why Are Large Crystals Preferred**?**

Because they are easy to wash and dry and less susceptible to contamination.

6- Filtration Under Reduced Pressure (Using a Buechner Apparatus)

7- Washing the Crystals

The washing process must be carried out to remove dissolved impurities trapped between the crystallized materials. Washing is done using a small amount of the selected solvent, and it must be cold.

8- Drying the Crystals

Activated Charcoal: A fine powder of charcoal prepared from burning bones. It is also called activated carbon.

Conditions That Must Be Followed When Adding Animal Charcoal in the Presence of Colored Impurities

1. Animal charcoal is added after ensuring that the organic substance has dissolved in the solvent.
2. Do not add a large amount of animal charcoal because excess amounts lead to the adsorption of part of the substance to be purified, causing a loss in product yield.
3. Animal charcoal is added while the solution is hot (the heating period should be short with shaking), and it should not be heated for a long period because the efficiency of activated carbon decreases with increasing temperature.

Procedure:

1. Weigh (1 g) of the impure substance in a (100 mL) beaker, then add (25 mL) of solvent and small pieces of boiling stones.

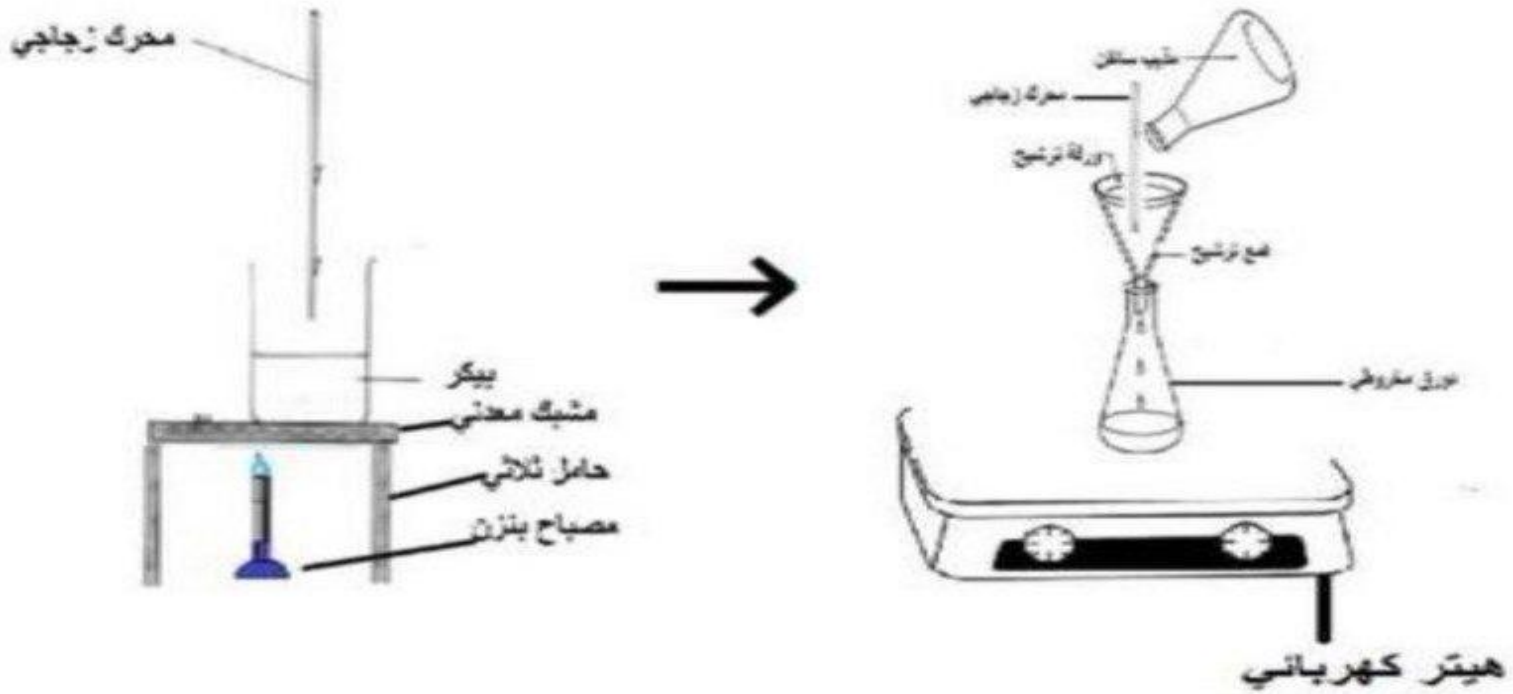
2. Heat the solution to boiling with continuous stirring using a glass rod until the substance dissolves.

3. Remove the beaker from the heat and add a small amount of animal charcoal, then allow the solution to boil for one minute.

4.Filter the solution while hot to remove insoluble impurities using a conical flask, a glass funnel, and folded filter paper. Wash the filter paper with a small amount of hot solvent, then cool the filtrate using an ice bath.

5.Filter the crystals after they appear using a Buechner funnel under reduced pressure, and wash the crystals with a small amount of cold solvent.

6.After completing the Buechner filtration, remove the filter paper containing the substance from the Buechner funnel and dry it in air by placing the paper on a watch glass. After drying well, weigh the precipitate and calculate the percentage yield of the substance.



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