The Islamic University

Department of Clinical Laboratory Sciences

Title of the course: *Practical Biochemistry* I

Level: 3rd Class, 1st Semester

Separation Techniques

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Experiment 3 : Recrystallization

The products of chemical reactions can be impure . Purification of your products must be performed to remove by-products and impurities . **Liquids** are customarily purified by **distillation** , while **solids** are purified by recrystallization (sometimes called simply "**Crystallization**") .

Recrystallization : is an important technique for purifying solid organic compounds.

It is based on the principle that solids are more soluble in hot solvents than in cold solvents.

The most important factors in a recrystallization are:

a) choosing a good solvent

b) using the right amount of solvent.

Characteristics of a Good Recrystallization Solvent:

1.The recrystallization solvent should NOT dissolve the substance to be purified at room temperature.

2.The solvent should dissolve soluble impurities well at room

.temperature

3. The solvent should not dissolve insoluble impurities even These insoluble impurities can then be removed by filtration.

4. The solvent must not react with the substance to be purified

5 .The solvent should be volatile.

6.The solvent should be inexpensive .

Note : Characteristics 1, 2, 3, and 4 are essential for a good recrystallization solvent Characteristics 5 and 6 are desirable, but **not** essential.

Solvent	Formula	Polarity	Boiling point (^{0}C)
Water	H_2O	Very polar	100
ethanol	CH_3CH_2OH	Polar	78
methanol	CH_3OH	Polar	65
dichloromethane	CH_2Cl_2	Slightly polar	40
diethyl ether	$(CH_3CH_2)_2O$	Slightly polar	35

Purpose of experimental : To separate organic compounds (solid) from impurities by recrystallization.

Chemical and Apparatus:

1. Erlenmeyer flask (125 mL)

2. short-stemmed funnel

3. Hot plate

4. boiling chips

5. benzoic acid

Procedure of Experimental

1. To perform a recrystallization, heat about 10 ml of the recrystallization solvent to boiling in a beaker in your hot plate use a stir bar to keep it from bumping.

2. Add the boiling solvent slowly to the (0.5 g) impurity benzoic acid (in a separate beaker) with a pipet, stirring it with a spatula and heating it on the hot plate until the solid has dissolved and the solvent is still boiling.

3. Filter the hot solution by gravity filtration using filter paper

4. Cool the solution to room temperature and then place the flask in ice water.

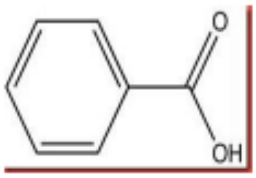
5. Wait more minutes and then collect the crystals using a vacuum filter apparatus. Wash the crystals with a very small amount of ice cold solvent.

6. Allow the crystals to dry with the vacuum on for several minutes. During this time, crystals may be observed forming in the mother liquor.

7. Determine the weigh the DRY crystals of recovered benzoic acid. Calculate the percent recovered using the following written formula and determine the melting point of your recrystallized benzoic acid.

$$\% \text{ Recovered} = \frac{\text{Weight of benzoic acid obtained after recrystallization}}{\text{Weight of benzoic acid before recrystallization}}$$

(Structure of benzoic acid)

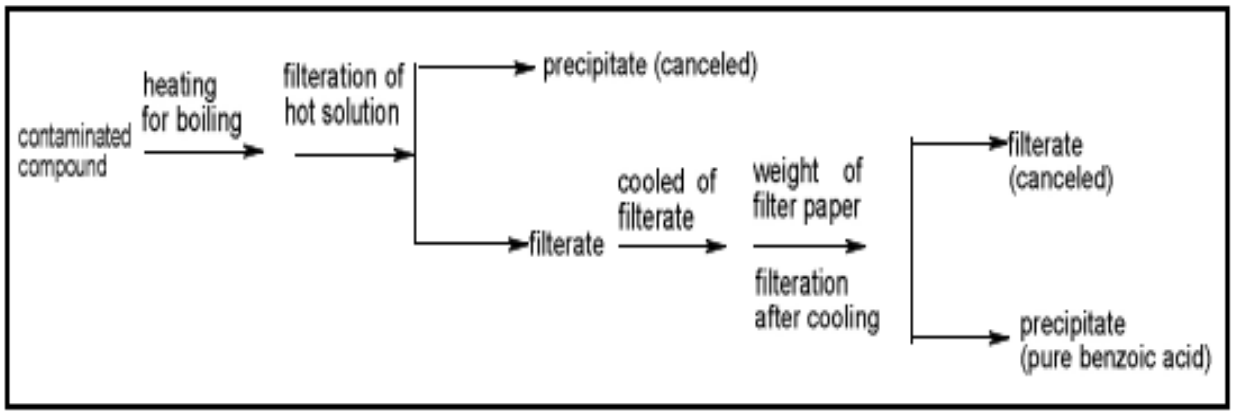


Questions for discussion

1. What is the ideal solvent for crystallization of a particular compound? What is the primary consideration in choosing a solvent for crystallizing a compound?

2. What is the purpose of recrystallization?

Plan of work


contaminated compound
heating for boiling
filteration of hot solution
precipitate (canceled)
filterate
cooled of filterate
weight of filter paper
filteration after cooling
filterate (canceled)
precipitate (pure benzoic acid)