

Recrystallization

Purpose of experimental

To separate organic compounds (solid) from impurities by recrystallization.

Theory part of experimental

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 compound is dissolved in the smallest possible amount of boiling solvent and then the solvent is allowed to cool, and the compound precipitates out. Hopefully, the impurities remain in the cold solvent, and only the pure compound precipitates out.

The most important factors in a recrystallization are:

- a) choosing a good solvent
- b) using the right amount of solvent.

If the solvent polarity is too similar, the compound will dissolve. If the solvent polarity is too different, the compound won't dissolve at all. If the solvent polarity is moderately different, then temperature will have a big impact on the solubility, allowing the compound to dissolve in boiling solvent but crystallize out when the solvent is cooled. Getting the right amount of solvent is a matter of proper technique.

Characteristics of a Good Recrystallization Solvent:

1. The recrystallization solvent should NOT dissolve the substance to be purified at room temperature, but it should dissolve it well at the solvent's boiling point
2. The solvent should dissolve soluble impurities well at room temperature.
3. The solvent should not dissolve insoluble impurities even at the solvent's boiling point. These insoluble impurities can then be removed by gravity 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.

Commonly Used Solvents

Solvent	B.P.	Dielectric Constant*	Safety Concerns
Water	100	78.4	None
Ethanol	78	24.5	Flammable; irritating to the eyes, respiratory system, and skin
Acetone	56	20.6	Flammable; Irritating to the eyes; Vapors may cause drowsiness or dizziness

Commonly Used Solvents

Solvent	B.P.	Dielectric Constant*	Safety Concerns
Tetrahydrofuran	66	7.58	Flammable; Harmful if swallowed; Irritating to the eyes, skin, and respiratory system
Ethyl acetate	78	6.02	Flammable; Irritating to the eyes; Vapors may cause drowsiness or dizziness
Cyclohexane	81	1.89	Flammable; May damage lungs if swallowed; Vapors may cause drowsiness or dizziness

*The dielectric constant is a measure of the solvent's ability to separate ions. In general, ionic compounds are more soluble in solvents with high dielectric constants.

Chemical and Apparatus

Erlenmeyer flask (125 mL), short-stemmed funnel, hot plate, boiling chips, 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.

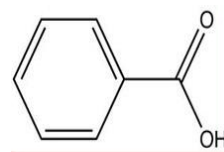
Experimental No.(4)

Recrystallization

- Cool the solution to room temperature and then place the flask in ice water.
- 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.
- Allow the crystals to dry with the vacuum on for several minutes. During this time, crystals may be observed forming in the mother liquor.
- 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.

$$\% \text{ Recovered} = \frac{\text{Weight of benzoic acid obtained after recrystallization}}{\text{Weight of benzoic acid before recrystallization}} \times 100$$

(Structure of benzoic acid)



Questions for discussion

- What is the ideal solvent for crystallization of a particular compound? What is the primary consideration in choosing a solvent for crystallizing a compound?
- What is the purpose of recrystallization?

Plan of work

