

# Chapter 12: Recrystallization

Recrystallization is a technique that chemists use to purify solid compounds. It is one of the fundamental procedures each chemist must master to become proficient in the laboratory. Recrystallization is based on the principles of solubility: compounds (solutes) tend to be more soluble in hot liquids (solvents) than they are in cold liquids. If a saturated hot solution is allowed to cool, the solute is no longer soluble in the solvent and forms crystals of pure compound. Impurities are excluded from the growing crystals and the pure solid crystals can be separated from the dissolved impurities by filtration.

This simplified scientific description of recrystallization does not give a realistic picture of how the process is accomplished in the laboratory. Rather, successful recrystallization relies on a blend of science and art; its success depends more on experimentation, observation, imagination, and skill than on mathematical and physical predictions. Understanding the process of recrystallization in itself will not make a student a master crystallizer; rather, this understanding must be combined with laboratory practice to gain proficiency in this technique.

## 12.1 How Recrystallization Works

You are likely familiar with a tasty demonstration of recrystallization: the formation of “rock candy.” Rock candy is made by dissolving sugar in hot water to the point of saturation. The clear solution is allowed to cool and evaporate very, very slowly. Days later, beautiful large crystals of pure sugar have grown in the liquid.

Three terms are important to know for this process: solvent, solute, and solution. The following definitions are taken directly from the CRC Handbook of Chemistry and Physics:

- Solvent: “That constituent of a solution which is present in larger amount, or, the constituent which is liquid in the pure state, in the case of solutions or solids or gasses in liquids.”
- Solute: “That constituent of a solution which is considered to be dissolved in the solvent. The solvent is usually present in a larger amount than the solute. A solution is saturated if it contains at a given temperature as much of a solute as it can retain in the presence of an excess of that solute.”
- Solution: “The word solution is used to describe a liquid or solid phase containing more than one substance, when for convenience one of the substances, which is called the solvent and may itself be a mixture, is treated differently from the other substances, which are called solutes.”

In the case of rock candy formation, the solvent would be water, the solute would be sugar, and the solution would be the sugar-water.

Recrystallizations in the organic laboratory proceed in a similar manner to the formation of rock candy (Figure 12-1). Just enough hot solvent is added to a small amount of an impure, solid compound in a flask to completely dissolve it. The flask then contains a hot solution, in which solute molecules – both the desired compound and impurities – move freely among the hot solvent molecules. As the solution cools, the solvent can no longer “hold” all of the solute molecules, and they begin to leave the solution and form solid crystals. During this cooling, each solute molecule in turn approaches a growing crystal and rests on the crystal surface. If the geometry of the molecule fits that of the crystal, it will be more likely to remain on the crystal than it is to go back into the solution. Therefore, each growing crystal consists of only one type of molecule, the solute. After the solution has come to room temperature, it is carefully set in an ice bath to complete the recrystallization process. The chilled solution is then filtered to isolate the pure crystals and the crystals are rinsed with chilled solvent.

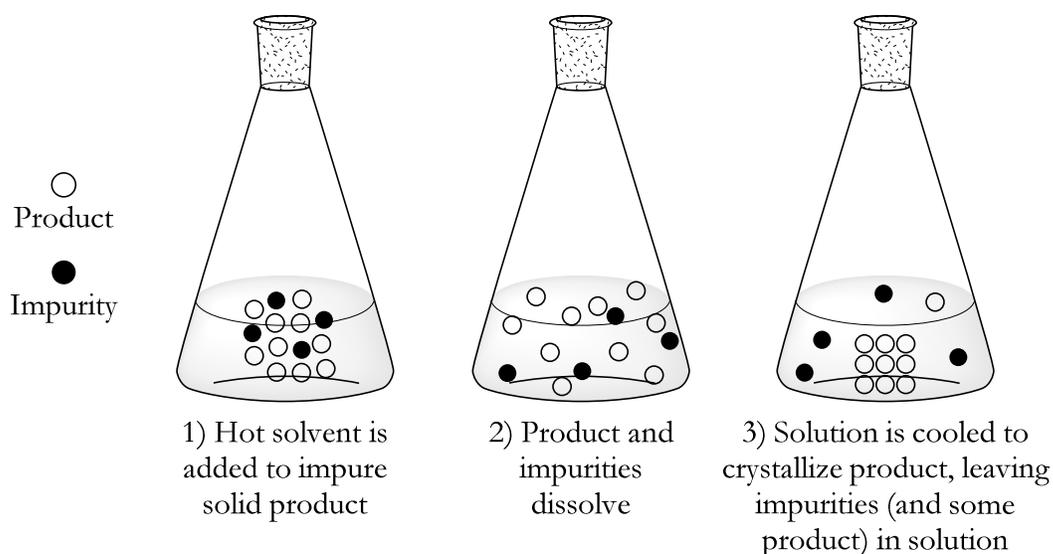


Figure 12-1: The general scheme for recrystallization.

Although the terms “crystallization” and “recrystallization” are sometimes used interchangeably, they technically refer to different processes. Crystallization refers to the formation of a new, insoluble product by a chemical reaction; this product will then precipitate out of the reaction solution as an amorphous solid containing many trapped impurities. Typically, crystallization is the method used to obtain the crude product. Recrystallization does not involve a chemical reaction; the crude product is simply taken into solution, and then the conditions are changed so as to allow crystals to re-form. Recrystallization produces a much purer, final product. For this reason, experimental procedures that produce a solid product by crystallization normally include a final recrystallization step to give the pure compound.

Recrystallization is not generally thought of as a separation technique; rather it is a purification technique in which a small amount of an impurity is removed from a compound. However, if the solubility properties of two compounds are sufficiently different, recrystallization can be used to separate them, even if they are present in nearly equal amounts.

Recrystallization is not a perfect technique. A percentage of compound is always lost because some of the compound will be soluble in cold solvent. If there is a lot of an impurity with the same solubility properties as the compound in which you are interested, the recrystallization will not yield pure compound. Sometimes a chemist simply cannot find a solvent with the proper solubility characteristics to recrystallize a particular compound or to prevent oiling out during the process. This is a common problem when working with low-melting compounds.

## 12.2 How to Choose a Recrystallization Solvent

A successful recrystallization depends on the proper choice of solvent. The compound must be soluble in the hot solvent and insoluble in the same solvent when it is cold. For the purpose of recrystallization, we consider 3% the dividing line between soluble and insoluble: if 3 g of a compound dissolves in 100 mL of a solvent, it is considered soluble, if not, it is considered insoluble. Table 12-1 contains hypothetical solubility data. In choosing a solvent, the bigger the difference between hot solubility and cold solubility, the more product will be recoverable from a recrystallization.

Study Table 12-1 and convince yourself that the following statements are true:

- Compound A is soluble in boiling water but not in cold water, therefore water would be a good choice for a solvent to recrystallize the compound.
- Compound A is not soluble in either hot or cold hexanes; hexanes would be a poor choice to recrystallize this compound.
- Compound B could not be recrystallized from water, although ethanol would be a good choice.
- Compound B is soluble in hexanes both at high and at low temperatures, therefore it could not be recrystallized from hexanes.
- Compound C could be recrystallized from methanol but not from water.
- If Compound C were recrystallized from ethanol, the yield would be low.

Table 12-1: Hypothetical solubility data.

Compound	Solvent	Solubility in Boiling Solvent	Solubility in Solvent at 0°C
A	Water	4 g/100 mL	0.2 g/100 mL
A	Hexanes	0.1 g/100 mL	0.0002 g/100 mL
B	Water	0.3 g/100 mL	0.02 g/100 mL
B	Hexanes	10 g/100 mL	9 g/100 mL
B	Ethanol	3 g/100 mL	0.1 g/100 mL
C	Water	4 g/100 mL	3 g/100 mL
C	Methanol	3 g/100 mL	0.3 g/100 mL
C	Ethanol	4 g/100 mL	2.8 g/100 mL

Determining an appropriate recrystallization solvent might be a trivial process if extensive solubility data for all known organic compounds was listed in easy-to-read and readily available tables. This is not the case. Sometimes solubility data for a particular compound can be found in a laboratory text, the scientific literature, or the Merck Index. The CRC (Table C) only lists *qualitative* values for the solubility of organic compounds. Therefore, determination of a solvent with the appropriate solubility parameters for a particular compound usually is made by a trial-and-error process.

Where do you begin this trial-and-error process of finding a suitable recrystallization solvent? First, consider the rule of thumb: “like dissolves like” (Table 12-2).

A polar compound dissolves in a polar solvent and a non-polar compound in a non-polar solvent. Try one solvent and if it does not work, try another. The process is not always trivial and it is not always predictable.

In the undergraduate organic chemistry teaching laboratories at CU, you will usually be told which solvent to use to recrystallize your product compounds. These recrystallization choices were determined by a process of trial and error, as in the sample procedure given below.

Table 12-2: Best choice of solvent for recrystallizing various types of compounds.

Compound type	Solvents in which the compound is likely to be soluble
Hydrocarbon	Petroleum ether, hexanes, cyclohexane, toluene
Ether	Diethyl ether, methylene chloride
Halide	Dichloromethane, chloroform
Carbonyl	Ethyl acetate, acetone
Alcohol, acid	Ethanol
Amine	Ethanol, water

### A. Procedure for Determining a Recrystallization Solvent

Place about 50 mg of the sample in a test tube. Add about 0.5 mL of cold solvent; if the sample dissolves completely, the solubility in the cold solvent is too high to be a good recrystallization solvent. If the sample does not dissolve in the cold solvent, heat the test tube until the solvent just boils. If the sample has not completely dissolved at this point, add more boiling solvent dropwise until all the solid dissolves. If it takes more than 3 mL to dissolve the sample in the hot solvent, the solubility in this solvent is probably too low to make it a good recrystallization solvent. If your first choice of solvent is not a good recrystallization solvent, keep trying others. If you cannot find a single solvent that works, try a two solvent system (see section 9.3).

### B. Factors Other than Solubility to Consider

If you find more than one solvent that satisfies the solubility criteria, consider the toxicity, flammability, volatility, boiling point, and reactivity properties of the potential solvents (see Table 12-3).

Always choose the solvent with the lowest toxicity; solvents such as benzene, chloroform, carbon tetrachloride, dioxane, and pyridine are suspected or proven human carcinogens or mutagens and should be avoided whenever possible. Benzene can be substituted by the less toxic toluene; chloroform by the less toxic dichloromethane. For safety reasons, if possible choose a solvent that is nonflammable.

Solvents should not be volatile: they should not have low boiling points or evaporate easily. Solvents that boil over 50°C are preferred, otherwise they evaporate too quickly at room temperature and during filtration. Solvents that boil over 120°C are difficult to remove from the recrystallized product, because the final step in a recrystallization is the evaporation of solvent from the crystals, and this takes a very long time at room temperature if the solvent has a high boiling point.

The solvent must not react chemically with the product: acetone cannot be used to recrystallize primary or secondary amines because condensation products may result; ethyl acetate cannot be used for alcohols due to possible esterification of the alcohol if traces of acid are present; acetic acid cannot be used with acid labile substances.

Table 12-3: Properties of common solvents (listed in order of decreasing polarity).

Solvent	Melting pt (°C)	Boiling pt (°C)	Density (g/mL)	Water miscible	Flammability	Health hazards
Water	0	100	1.00	+	none	none
Methanol	-98	65	0.79	+	moderate	poison
Ethanol	-114	78	0.81	+	moderate	irritant
Acetic acid	17	118	1.05	+	low	corrosive
Acetone	-94	56	0.79	+	high	irritant
Dichloro- methane	-97	41	1.34	-	none	irritant
Ethyl acetate	-84	77	0.90	-	moderate	irritant
Chloroform	-63	61	1.48	-	none	suspected carcinogen
Diethyl ether	-116	35	0.71	-	very high	irritant
Toluene	-93	111	0.87	-	moderate	irritant, poison
Benzene	5	80	0.88	-	high	carcinogen
Petroleum ether	-35	30-60	0.63	-	high	irritant
Ligroin	-45	60-80	0.68	-	high	irritant
Hexanes	-95	69	0.66	-	high	irritant

The boiling point of the solvent should be lower than the melting point of the compound to be recrystallized. Otherwise, the solid melts before the solvent boils and can separate as oily droplets that are difficult to get into solution. This process is called “oiling out.”

Another consideration in the choice of a solvent is the solubility of impurities. The impurities, which you need to remove from your compound, must be either insoluble in the hot solvent or soluble in the cold solvent. Otherwise, they will crystallize along with your compound. If they are insoluble in the hot solvent, they are filtered away in a hot filtration. If they are soluble in the cold solvent, they remain in solution when the desired compound crystallizes.

### C. Summary

- The compound must be soluble in the hot solvent and insoluble in the cold solvent.
- The solvent should be nontoxic, noncarcinogenic, and nonflammable, non-volatile.
- The solvent should boil in the range 50–120°C.
- Impurities should either be insoluble in the hot solvent or soluble in the cold solvent.
- The solvent must not react with the compound.
- The boiling point of the solvent should be lower than the melting point of the compound.

### D. Procedure for Recrystallization

#### 1. Dissolve the sample in hot solvent.

Place the compound to be recrystallized in an Erlenmeyer flask. (This is a better choice than a beaker, since the sloping sides help trap solvent vapors and slow the rate of evaporation.) Heat the solvent in a separate flask to its boiling point on a hotplate. (Remember to add boiling chips while the solvent is still cool, to keep it boiling smoothly). Add hot solvent to the flask containing the compound in small portions, swirling after each addition, until the compound is completely dissolved. Keep the solution hot at all times during the dissolution process by resting it on the hotplate too. Do not add more hot solvent than is necessary to just dissolve the sample.

There is no set “rule” as to how much hot solvent you need to add per gram of compound. We cannot tell you to add X mL of solvent to Y g of compound. You simply add as much hot solvent as it takes in each individual experiment.

If a portion of the solid does not seem to dissolve even after more hot solvent has been added, it is likely due to the presence of very insoluble impurities. If this happens, stop adding solvent and do a hot filtration before proceeding.

If the solution is colored and you suspect colored impurities are present, treat the hot solution with Norite.

#### 2. Cool the solution.

Once the compound is completely dissolved in the hot solvent, and colored or insoluble impurities have been removed if necessary, the cooling and recrystallization phase can begin.

The rate of cooling determines the size and quality of the crystals: rapid cooling favors small crystals and slow cooling favors the growth of large and generally purer crystals. In the student laboratory, time is usually at a premium. A convenient compromise between speed of recrystallization and quality of crystals is to set the flask containing the dissolved compound on a surface that does not conduct the heat away too quickly, such as a paper towel set on the benchtop. Leave the flask undisturbed until it comes to room temperature. The rate of recrystallization is usually greatest at about 50°C below the melting point of the substance; the maximum formation of crystals occurs at about 100°C below the melting point.

It is a good idea to lightly cover the flask as it cools to prevent evaporation and to prevent dust from falling into the solution.

Observe the flask as it comes to room temperature: you should see crystals begin to form. If they do not form, try some of the suggestions in the section, “Common Problems Encountered in Recrystallization.”

Once the crystals have formed, place the solution in an ice bath to ensure that the maximum amount of crystals are obtained. The liquid in the flask above the crystals is called the *mother liquor*. Note that the mother liquor still contains some of the compound dissolved in the now cold solvent.

### 3. Isolate the crystals.

Isolate the crystals by vacuum filtration. Rinse the crystals on the Büchner funnel with a small amount of fresh, cold solvent to remove any impurities that may be sticking to the crystals. The rinse solvent is always the same solvent that was used to crystallize the compound. If you used a mixed solvent, use a wash solvent composed of the same two solvents in approximately the same percentages as used for the recrystallization.

The filtrate, or the mother liquor, may contain a significant amount of compound. To obtain a higher yield of product, you can collect a second batch of crystals, known as the *second crop*. To do this, concentrate the mother liquor by boiling off some of the solvent and then allowing the solution to cool and crystallize as before. Oftentimes, the second crop is not as pure as the first crop since there is a greater percentage of impurities in the compound to begin with.

### 4. Dry the crystals.

The crystals are dried by leaving them undisturbed on the filter funnel and drawing air across them for several minutes. If the solvent is low-boiling and volatile, this is sufficient in most cases to dry the crystals.

Crystals can also be air-dried by allowing them to stand uncovered for several hours or days. More efficient methods include vacuum drying or placing in a desiccator.

## E. Common Problems Encountered in Recrystallization

Certain factors cannot always be controlled in a recrystallization: the amount of impurities, the nature of impurities, and the availability of a suitable recrystallization solvent. Even if these factors have been optimized, the beginning organic chemist often makes mistakes in judgment during a recrystallization that lead to either low yields of crystals or the isolation of impure crystals.

### 1. Crystals do not form when the solution is cooled.

If the hot solution is supersaturated with compound, as the solution cools, it may enter a supercooled state wherein the crystals *would* precipitate but do not have a nucleation site on which to start forming. If this occurs, several methods can be tried to induce recrystallization, each of which provides a nucleus for the recrystallization to begin.

- Scratch the side of the glass flask; this loosens small particles of glass that act as nucleation points
- Cool the solution in an ice bath
- Add a seed crystal (a crystal of pure compound)

If none of these works, you have added too much solvent and need to reduce the volume of the solution by boiling it down to about half its current volume, then try cooling the solution again.

## 2. The compound forms an oil instead of a solid.

The phenomenon known as “oiling out” is the separation of the compound as an oil rather than as crystals – instead of crystals, you see an “amorphous blob” on the bottom of the flask. Oiling out can occur in these situations:

- The melting point of the compound is lower than the boiling point of the solvent
- The solute is contaminated with impurities, effectively lowering the melting point of the solute (this is more likely to occur when a compound to be recrystallized is very impure)

Since the formation of an oil is not a selective process, oiling out is not an effective means of purifying a compound. If it does occur, try some of the techniques listed below to obtain crystals of the compound:

- Redissolve by heating the solution to a temperature lower than the boiling point of the solvent, then try scratching the sides of the flask or seeding with pure crystals during the cooling process, and allow it to cool very, very slowly
- Add more hot solvent, since diluting the mixture can help
- Isolate the oil and redissolve it in hot solvent, either the same or a different one
- Reattempt the recrystallization using a different solvent, perhaps one with a lower boiling point
- Repurify the compound by another method, such as a chromatographic technique, before the recrystallization

## 3. The crystals dissolve when they are rinsed with solvent during filtration

After isolating the crystals by filtration, they should be rinsed with a small amount of cold solvent to remove traces of impurities. If they are rinsed with solvent that is too warm, they will redissolve into the solvent. Always use a fresh portion of the same solvent that was used to recrystallize the compound rather than a random solvent off the shelf, because you do not know the solubility properties of the compound in the “random” solvent.

## 4. The recovered product is not of high purity.

In a recrystallization, solute molecules move reversibly between a growing crystal and the solution. The crystals select only those molecules that fit into its structure, declining impurity molecules that do not fit. However, if the solution is cooled too quickly, *precipitation* occurs. In a precipitation, the crystals are formed so rapidly that impurities are trapped in the crystal lattice. The small crystals of precipitates also have a large surface area on which even more impurities can adhere. If your melting point indicates impurities or your crystals are small or not pure in appearance, repeat the process using a longer period of cooling.

The result of disturbing the flask during the period of crystal growth is similar to that of cooling too quickly. Precipitation rather than recrystallization occurs, resulting in small crystals and trapped impurities.

## 12.3 Other Recrystallization Techniques

### A. Mixed Solvent Recrystallization

Sometimes no single solvent possesses the desired solubility properties: soluble in hot solvent and insoluble in cold solvent. In this case, a mixed solvent system may be used. A mixed solvent consists of

two miscible solvents, one in which the substance is soluble, and the other in which the substance is insoluble.

In a mixed solvent recrystallization, the compound is dissolved in the minimum amount of the hot solvent in which it is soluble, and then small amounts of the hot solvent in which it is not soluble are added to the hot mixture. The mixture is kept hot and the hot solvent in which it is not soluble is added dropwise until the mixture appears cloudy. Then more of the solvent in which it is soluble is added to clear the mixture resulting in a hot, saturated solution of the compound in a *mixed* solvent. When the solvent is allowed to cool, crystals of the compound will form. The two solvents must be miscible with each other and of significantly different polarities. Table 12-4 lists some common solvent pairs.

Table 12-4: Compatible solvent pairs.

Ethanol & water	Diethyl ether & methanol	Dichloromethane & methanol
Methanol & water	Diethyl ether & acetone	Diethyl ether & hexanes
Acetic acid & water	Ethanol & acetone	Chloroform & petroleum ether
Acetone & water	Ethanol & hexanes	Toluene & hexanes

Sometimes the proper solvent mixture is known before the recrystallization is performed. In this case, the mixed solvent is used in the same manner as “pure” solvent.

## B. Removal of Colored and Solid Impurities

If the sample is contaminated with impurities that are colored or impurities that remain solid when the compound is dissolved in hot solvent, the two methods below are used to separate them from the desired compound.

### 1. Removal of colored impurities by addition of Norite

Solid organic compounds frequently contain small amounts of colored impurities, enough to turn the solution yellow, brown, blue, etc. These impurities may be by-products from a synthesis or isolation process, or they may derive from oxidation and degradation reactions that occur when a compound is improperly stored.

The presence of colored impurities does not cause a problem as long as the colored impurities remain in solution when the compound crystallizes. However, the nature of colored impurities means that they are often adsorbed by the crystals as they form, resulting in an impure, colored product.

Colored contaminants are usually polar due to the presence of chromophore groups such as NO<sub>2</sub>, CN, and –N=N–. This property can be used to advantage to remove them from the solution before the recrystallization step, because their polarity causes them to adsorb readily to activated charcoal. Activated charcoal, sold under the trade name Norite, has a large surface area on which polar (and often colored) compounds are selectively adsorbed.

Norite is sold either as a fine powder or as small pellets. If the fine powder is used, the hot solution must be removed from the heat source before the Norite powder is added or the addition will cause the solution to bump and boil over the top of the flask. The fine Norite powder is then removed by hot filtration (see Chapter 10) before the solution is allowed to cool. If the Norite pellets are used, they are added to the hot solution and the solution is swirled for about 5 minutes until the color disappears; more pellets are added if necessary. During this process, the flask is kept hot. Then, the Norite pellets are removed by filtering or decanting the solution into a clean flask and the solution is allowed to cool.

## 2. Removal of solid impurities from hot solvent by hot filtration

If the compound dissolves in hot solvent and the impurities remain insoluble, a “hot filtration” is carried out to remove the impurities (see Chapter 10). In a hot filtration, the solution is gravity-filtered through a piece of fluted filter paper, taking care at all times to keep the flask and filter hot so that the desired compound does not begin crystallizing out of solution before the process is completed.

## 12.4 Study Problems

1. What is the purpose of a recrystallization?
2. You want to purify 10 g of Compound A that has been contaminated with 0.2 g of Compound B. Solubilities in water of the two compounds are given in the following table:

Compound	Solubility at 20°C (g/10 mL)	Solubility at 100°C (g/10 mL)
Compound A	0.029	0.680
Compound B	0.22	6.67

- a. What volume of boiling water is needed to dissolve the 10 g of Compound A?
  - b. How much Compound A will crystallize after cooling to 20°C?
  - c. Will any Compound B crystals also form?
  - d. What is the maximum amount of Compound A that can be recovered in the first crop of this recrystallization?
  - e. Will the Compound A be pure?
3. You want to purify 10 g of Compound A that has been contaminated with 10 g of Compound B. (Consider the table given in question 9.2.)
    - a. What volume of boiling water is needed to dissolve the 10 g of Compound A?
    - b. How much Compound A will crystallize after cooling to 20°C?

c. What amount of Compound B crystals will also form?

d. Will the Compound A be pure?

4. You have a sample of 0.1 g of compound C that is contaminated with a compound D. Compound C has the following solubility properties:

Solubility at 25°C	Solubility at 100°C
0.01 g/mL	0.1 g/mL

a. If compound C is completely insoluble in water and 2 mg of compound D is present, how could you purify Compound C?

b. If compound D has the same solubility behavior as C and 2 mg of this compound is present, how could you purify compound C? Would one recrystallization produce absolutely pure C?

c. Assume compound D has the same solubility behavior as C and 25 mg of this compound is present. Would one recrystallization produce absolutely pure C? How many recrystallizations would be needed to produce pure C? How much C would have been recovered when the recrystallizations have been completed? (If you include multiple recrystallizations in your answer, use just enough hot solvent in each step to completely dissolve compound C.)

5. Listed below are solubility vs temperature data for an organic compound in water.

Temp.	Sol. in 10 mL water
0	0.15 g
20	0.30 g
40	0.65 g
60	1.10 g
80	1.70 g

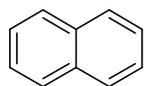
a. Using the data in the above table, graph the solubility of the compound vs temperature. Connect the data points with a smooth curve.

b. Suppose 0.1 g of this compound is mixed with 1.0 mL of water and heated to 80°C. Would all of the compound dissolve? Explain.

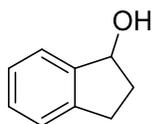
c. The solution prepared in (b) is cooled. At what temperature will crystals of the compound

appear?

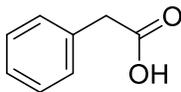
- d. Suppose the cooling described in (c) were continued to 0°C. How many grams of the compound would come out of solution?
6. You have 2 g of benzhydrol and have been advised to recrystallize it from hexanes. How much hexanes will you use to recrystallize this product?
7. Suppose you are recrystallizing a compound and boil the solution for so long that a substantial amount of the liquid evaporates. What is likely to happen to some of the solute? What should you do if this occurs?
8. Suppose you have prepared a compound that is reported in the literature to have a pale blue color. When dissolving the substance in hot solvent prior to recrystallization, the resulting solution is blue. Should you use decolorizing charcoal before allowing the hot solution to cool? Explain your answer.
9. Each of the following compounds, A–D, is equally soluble in the three solvents listed. In each case, which solvent would you choose? Explain.
- Compound A: benzene, acetone, or chloroform
  - Compound B: carbon tetrachloride, methylene chloride, ethyl acetate
  - Compound C: methanol, ethanol, or water
  - Compound D: ethanol, acetone, or diethyl ether
10. Suggest possible recrystallization solvents for the following compounds.



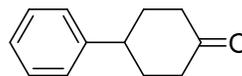
Naphthalene



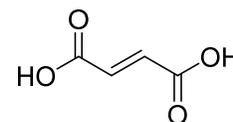
2-Indanol



Phenylacetic acid



4-Phenylcyclohexanone



Dimethyl fumarate

11. A student was recrystallizing a compound. As the hot solution cooled to room temperature, no crystals appeared. The flask was then placed in an ice-water bath. Suddenly a large amount of solid material appeared in the flask. The student isolated a good yield of product, however, the product was contaminated with impurities. Explain.
12. A student used benzene to recrystallize a compound. As the hot solution cooled to room temperature, very few crystals appeared. The flask was then placed in an ice-water bath. Suddenly a large amount of solid material appeared in the flask. Then, the student filtered the solid with vacuum, but only a few crystals remained on the filter paper. Explain these results.