

Technique F: Recrystallization, Filtration, and Sublimation

Introduction to Recrystallization

The physical property that is most useful for purification of solids is differential solubility in an appropriate solvent. When crystals form during a reaction or following an extraction, impurities may become trapped within the crystal lattice or upon the surface of the solid. Washing the crystals with cold solvent can remove adsorbed impurities from the surface, but this process cannot remove the trapped (occluded) impurities. To remove these by recrystallization, it is necessary to redissolve the solid in hot solvent, filter off any insoluble impurities, and then cool the solution to let the material crystallize again.

Theory

Organic solids are usually more soluble in hot solvent than in a comparable volume of cold solvent. In recrystallization, a saturated solution is formed by carefully adding an amount of hot solvent just necessary to dissolve a given amount of solid. A slight excess of solvent may be required for hot gravity filtration used in miniscale experiments. As the solution cools, the solubility of the solid decreases and the solid crystallizes. Unavoidably, some of the solid remains dissolved in the cold solvent, so that not all of the crystals dissolved originally are recovered.

In order to be successfully separated from the impurities in a certain solvent, the solid and the impurities should have differing solubilities. Impurities that remain undissolved in the solvent can be removed by hot gravity filtration of the solution prior to cooling. Impurities that are more soluble will remain in solution after the solid crystallizes. If the impurities and the compound have very similar solubility characteristics, repeated recrystallizations may be required or a different solvent may be required for purification. A crystal of the desired compound (called a "seed" crystal) can be added to the cooling solution to encourage crystallization if crystals fail to form initially.

Solvents for Recrystallization

Selecting a solvent is crucial for the successful recrystallization of an organic solid. An ideal recrystallization solvent should:

- dissolve all of the compound at the boiling point of the solvent.
- dissolve very little or none of the compound when the solvent is at room temperature.
- have different solubilities for the compound and the impurities.
- have a boiling point below the melting point of the compound, so that the compound actually dissolves, not melts, in the hot solvent.
- have a relatively low boiling point (60-100°C) so that the solvent is easily removed from the crystals and the crystals are easy to air dry.
- be nonreactive with the compound, nontoxic, and not have an offensive odor.
- be relatively inexpensive.

A good recrystallization solvent should possess many, if not all, of these properties. The importance of selecting an appropriate recrystallization solvent cannot be overstated.

The compound being recrystallized should dissolve in a reasonable amount of hot solvent but be insoluble in the cold solvent. Recall the adage, "**like dissolves like.**" Polar compounds will dissolve in polar solvents, but not in nonpolar solvents. The opposite is true for nonpolar compounds. If the polarities of the compound and the solvent are too similar, the compound will be readily soluble in that solvent and will not crystallize. The compound and the solvent cannot have radically different polarities, or the compound will not dissolve at all. This implies that the compound and the solvent should have somewhat different—but not totally different—polarities. It is obvious that an understanding of polarity is crucial to the selection of a recrystallization solvent. Polarity is determined by dipole moments of substances. Compounds that contain only carbon and hydrogen are nonpolar. This includes compounds such as alkanes, alkenes, alkynes, and aromatic compounds. Somewhat more polar are ethers and halogenated compounds, followed by ketones and esters. More polar yet are alcohols. This trend is summarized in Table 1F-1.

Table 1F-1. Classes of Organic Solvents and Examples Used for Recrystallization

Polarity	Class	Examples
Most Polar	alcohols	methanol, ethanol, 2-propanol
	ketones	acetone, 2-butanone
	halogenated alkanes	1,2-dichloroethane
	ethers	diethyl ether, tetrahydrofuran
	aromatic compounds	toluene
Least Polar	alkanes	hexane, petroleum ethers

Factors that influence polarity and solubility of an organic compound are molecular weight and proportion of hydrocarbon in the molecule. Compounds with higher molecular weight are less soluble in general than those with lower molecular weight with the same functional group. The higher the proportion of hydrocarbon, the less polar the compound.

Common recrystallization solvents listed in Table 1F-2 are arranged in order of decreasing solvent polarity, as measured by the dielectric constant ϵ , a measure of a solvent's ability to moderate the force of attraction between oppositely charged particles (1.0 = a vacuum).

Table 1F-2. Properties of Common Recrystallization Solvents

Solvent	b.p. (°C)	ϵ	Examples
Very polar:			
Water	100	78.5	Good for polar compounds, difficult to remove from crystals
Polar:			
Methanol	65	33.0	Sometimes used instead of ethanol
Ethanol	78	24.3	Usually 95%, good for relatively nonpolar compounds
Acetone	56	21.2	Low boiling (2-butanone higher)
2-Propanol	82	18.3	Good for nonpolar compounds
Moderately polar:			
Ethyl acetate	77	6.0	Good for nonpolar compounds
Nonpolar:			
Toluene	110	2.4	High b.p., so hard to evaporate
Petroleum ether	90-110	2.0	Highly flammable, easy to evaporate
Petroleum ether	60-90	2.0	Highly flammable, easy to evaporate
Cyclohexane	81	1.9	Good for less polar compounds
Hexanes	69	1.9	Good for less polar compounds

Chemical reference books, such as the *CRC Handbook of Chemistry and Physics* or the *Merck Index*, are sources of valuable information about solubility. The abbreviations that are used are "i" (indicating that the compound is insoluble), "s" (soluble), "δ" (slightly soluble), "h" (soluble in hot solvent), and "v" (very soluble). A solvent that is listed as slightly soluble or soluble in hot solvent should be a good recrystallization solvent for that compound. Sometimes the handbook will give the exact solubility of a compound in a given solvent. The units of solubility are typically given as grams of compound dissolved per 100 mL of solvent. Unless otherwise noted, the solubilities are measured at 25°C.

The best way to be certain that a solvent will be a good recrystallization solvent is to try it and see! Test the solvent on a small amount of the compound. If that solvent doesn't dissolve the crystals when hot, or if it dissolves the crystals at room temperature, try another solvent. Keep trying until you find a solvent that dissolves the compound when the solvent is hot, but not when the solvent is cold.

Since it is usually easier to use a single solvent than a solvent pair, it is well worth the time and effort to try to find a single solvent for recrystallization. However, sometimes it happens that no solvent is found that will dissolve the compound when hot, but not when cold. In this case, a solvent pair must be used. A solvent pair consists of two miscible solvents, which have rather different polarities. A solvent pair then consists of two solvents, one in which the compound is soluble, the other in which the compound is insoluble. **The most common solvent pair is ethanol-water.**

Recrystallization using a single solvent is done by dissolving the compound in the **minimum amount of hot solvent**, filtering any insoluble impurities, then letting the

solution cool to room temperature. The rate at which the crystals form affects the purity of the crystals. Cooling too rapidly (such as immersing the warm solution in an ice bath) results in the formation of very small crystals that adsorb impurities from the solution. If crystals are too large, solution and impurities can be trapped (occluded) within the crystal lattice. The optimal crystals are obtained by leaving the flask undisturbed until crystallization occurs. When crystals do appear, the mixture should be cooled further in an ice bath to ensure complete crystallization. The crystals are then suction-filtered, rinsed with several small portions of ice cold solvent to remove traces of surface impurities, and allowed to dry.

Frequently, a second crop of crystals can be obtained from the filtrate (called the mother liquor). This entails heating the filtrate solution to reduce the volume, then letting the solution cool slowly as crystallization occurs. The crystals obtained from this second crop are usually not as pure as the initial crop. Melting points should be obtained and the purity evaluated before the two crops of crystals are combined. Since impurities usually lower the melting point and increase the melting point range, the measured melting points are good indications of the purity of the crystals.

The purity of the crystals is one measure of the success of a recrystallization procedure. A second measure is the percent recovery, that is, how many grams of pure compound are obtained relative to the amount of the impure crystals. The percent recovery for a recrystallization process can be determined by measuring the mass of the crystals before recrystallization and after. The percent recovery is given by the equation below.

$$(\text{mass of pure crystals} / \text{mass of impure crystals}) \times 100 = \text{Percent Recovery}$$

Low percent recoveries may be due to using the wrong solvent, using too much solvent, incomplete crystallization (not enough time or low enough temperature), or inefficient filtration technique. Since the compound usually has some solubility in the cold solvent, there is always some loss of product.

Choosing a Solvent

The first step (and most important step) is choosing a recrystallization solvent or solvent pair. This is necessary whether doing a microscale or a miniscale recrystallization.

General Procedure

You should narrow the choice of solvents by considering the polarity of the compound being recrystallized. For nonpolar compounds, try solvents of moderate polarity. For moderately polar compounds, try solvents of higher polarity, such as water. For polar compounds, try water or solvents of low to moderate polarity. (Refer to Table 1F-2 for solvent polarities.)

Place about 50 mg of the solid (about the tip of a spatula) into a small test tube. Add 1 mL of the solvent to be tested. Mix the contents thoroughly and observe. If most of the solid dissolves at room temperature, the compound is soluble in this solvent and it will not be a good recrystallization solvent. Start over with a new portion of the solid and a new solvent.

If, on the other hand, most of the solid does not dissolve at room temperature, gently heat the test tube to boiling in a sand bath or water bath. Observe.

- If the solid dissolves in the hot solvent, it will be a good recrystallization solvent.
- If only some of the solid dissolves, try adding another 1 mL portion of the solvent and heating in a sand bath or water bath. If more of the solid dissolves, the compound is slightly soluble and this solvent may work as a recrystallization solvent. Add enough **hot** solvent to dissolve all of the crystals. Then place the test tube in an ice bath. If many crystals form, this solvent will be good for recrystallization.
- If the solid does not at least partially dissolve in the hot solvent, the compound is insoluble in the hot solvent and the solvent will not be a good recrystallization solvent. Start over with a new portion of the solid and a new solvent.

This process is illustrated in Figure 1F-1.

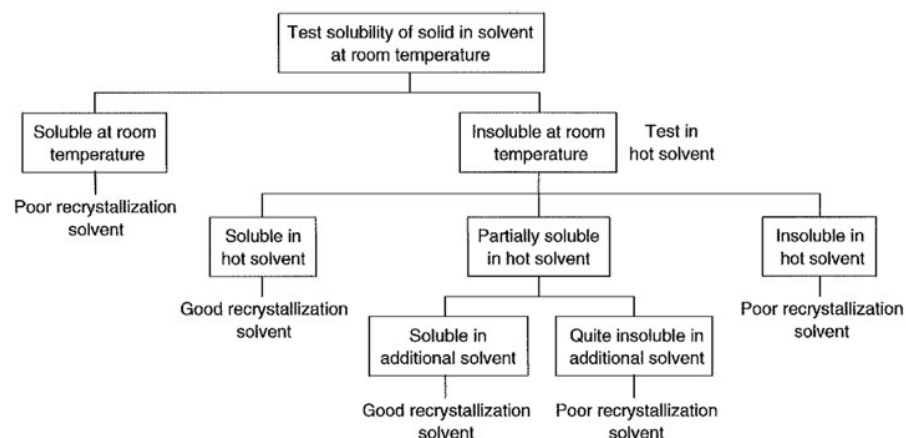


Figure 1F-1. Flow scheme for determining the suitability of a solvent for recrystallization of an organic solid.

Choosing a Solvent Pair

If no solvent is found that will dissolve the compound when hot but not cold, you must perform a solvent-pair recrystallization. This is easier to do if accurate and detailed notes have been recorded. Select a solvent that dissolved the compound and a different solvent that failed to dissolve the compound. **Remember that the two solvents selected must be miscible.** This means they mix in all proportions, such as ethanol and water. To ensure that the two solvents selected are miscible, put 1-mL portions of each solvent in a test tube and shake. If the solution is homogenous (one phase, clear), the solvents are miscible. If two liquid phases appear, the solvents are immiscible: they cannot be used for a solvent-pair recrystallization.

To test the solvent pair, place about 100 mg of the solid in a test tube. Add dropwise the hot solvent in which the compound is more soluble. Add just enough hot solvent to dissolve the solid, no more. Then add dropwise the hot solvent in which the compound is less soluble. If the solution turns cloudy due to the crystallization of the solid, this will be an acceptable solvent pair to use for recrystallization. Once the solvent or solvent system has been determined, proceed with the recrystallization process.

How to Do a Microscale Recrystallization

Microscale Single-Solvent Recrystallization

Microscale single-solvent recrystallization should be used to recrystallize 50 mg to 300 mg of material. Put the crude crystals into a 10-mL Erlenmeyer flask that contains a boiling chip. With a pipet, add approximately 0.2-0.3 mL aliquots of the chosen hot solvent. After each addition of solvent, swirl the crystals and heat the solution on a warm sand bath or heat block. Continue to add the hot solvent until the entire solid has dissolved. Be patient. Don't heat the solution too strongly or the solvent will boil away. Adjust the temperature of the heat source to keep the solution just under the boiling point of the solvent. If there is still undissolved solid, skip ahead to the next paragraph. Otherwise; continue. When the solid has dissolved, remove the Erlenmeyer flask from the sand bath and set it aside to cool to room temperature undisturbed. A large beaker packed with a paper towel or packing material works well to insulate the flask. Place the Erlenmeyer flask down into the beaker. After 10-15 minutes or when the flask is at room temperature, place the flask in a beaker filled with ice. Let the flask stand 5-10 minutes or until the flask feels cold to the touch. Place a small beaker of solvent in the ice bath to cool. If crystallization does not occur even after the solution is cold, scratch the inside of the flask with a glass rod or add a crystal of product (a seed crystal) to the solution. If crystallization still does not occur, reduce the volume of solution and let it cool to room temperature. After crystallization is complete, suction filter the crystals using a Hirsch funnel and small filter flask. Rinse the crystals sparingly with several drops of the ice-cold solvent and air dry the crystals.



If further addition of the hot solvent does not dissolve more of the solid, the solid particles

are probably insoluble impurities. If this is the case, it is necessary to filter the mixture. Prepare a Pasteur filter pipet (a Pasteur pipet fitted with a cotton plug). Hold the pipet over the flask for a minute or two to warm (to prevent crystallization within the pipet). Obtain a clean 10-mL Erlenmeyer flask or a 5-mL conical vial. Add 5-10 drops of the hot solvent to the flask or vial and set it on the warm sand bath or heat block. Squeeze on the bulb to remove the air and place the pipet in the mixture. Slowly release the bulb to draw up the solution into the Pasteur pipet. Try not to draw up any of the solid impurities. Transfer the solution to a clean flask or vial. Any solid that gets drawn up in the pipet will stick to the cotton. (See Figure 1F-4.) The pipet may be rinsed with one or two small portions of solvent. Sometimes crystals form immediately upon contact with the container. If this happens, add more hot solvent until all of the solid dissolves. Then remove the container from the heat source, allow it to cool to room temperature, and proceed as above.

Hot filtration should also be done if the solution is highly colored (when the pure compound is white!). In this case, it will be necessary to add a small amount of decolorizing carbon (such as Norit or charcoal) to the warm (not boiling!) solution. Decolorizing carbon has a very large surface area that enables it to adsorb colored impurities. To use decolorizing carbon, add 5-6 more drops of solvent and let the solution cool a little below the boiling point so as to avoid splattering. Add a spatula-tip full or less of the decolorizing carbon and swirl. Heat the black mixture for several minutes, then filter using a filter pipet. Transfer the clear solution to a clean container and proceed as described above. If small particles of carbon can be seen, it may be necessary to refilter using a new Pasteur pipet.

Microscale Solvent-Pair Recrystallization

This procedure should be used if no single solvent is found that will work for the recrystallization procedure.

Heat two small flasks, each containing one of the solvents to be used for the recrystallization. Place the crystals in a 10-mL Erlenmeyer flask or 5-mL conical vial. With a pipet, add dropwise the hot solvent that was found to best dissolve the compound. Swirl after each addition of hot solvent. When the crystals are dissolved, add dropwise the hot solvent that does not dissolve the compound. Observe carefully after each addition. Continue adding the solvent until the solution appears cloudy or crystal formation is observed. This is called the "cloud point." Then add 1-2 drops more of the first solvent to just redissolve the crystals and cause the cloudiness to disappear. Remove the container from the heat source and allow to cool to room temperature, undisturbed. Chill further in an ice bath, then suction filter the crystals. Wash with several small portions of ice-cold solvent (the solvent in which the compound is insoluble!). Air dry the crystals or place in a drying oven.

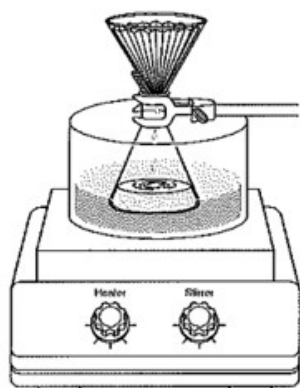
If some of the solid does not dissolve during addition of the first hot solvent, remove the solid impurities by drawing up the solution with a Pasteur filter pipet. Transfer the solution to a clean vial or Erlenmeyer flask. Add a little more of the first hot solvent if necessary. Then add the second hot solvent dropwise until the solution appears cloudy.

If no crystals are obtained upon cooling, reheat the solution. Again add dropwise the hot solvent that does not dissolve the compound. At the cloud point, cool the solution and wait for crystallization.

How to Do a Miniscale Recrystallization

Miniscale Single-Solvent Recrystallization

This procedure should be used for recrystallizing 300 mg or more of a solid. Transfer the impure solid into a 10-mL or larger Erlenmeyer flask. Heat the solvent on a hot sand bath or steam bath in the hood. Most organic solvents are flammable, so use extreme caution when heating. Add the minimum amount of the hot solvent required to dissolve the crude crystals. Swirl to dissolve, while heating. Continue to add small portions of the hot solvent until all of the solid dissolves. If it becomes apparent that insoluble impurities are present, any solids that do not dissolve may be removed by hot gravity filtration. Obtain a second Erlenmeyer flask and add 1-2 mL of the hot solvent. Set a stemless funnel fitted with a piece of fluted filter paper (see Figure 1F-6) on top of the Erlenmeyer. Set the funnel in an iron ring or place a paper clip between the funnel and neck of the Erlenmeyer. Heat the flask and funnel



so hot vapors fill the system. To keep the solid in solution, add 1-2 mL of the hot solvent to the first Erlenmeyer flask. Add a spatula-tip full of decolorizing carbon if the solution is deeply colored. Keeping everything hot, pour the solution through the filter paper in small batches. Continue pouring until all of the solution is transferred. The solution should be clear and colorless. If crystallization occurs in the flask or filter paper, add more of the hot solvent to dissolve the crystals. When all the solid is dissolved, remove the solution from the heat and let it stand undisturbed. Cool to room temperature (about 15-20 minutes). After crystallization occurs, place the flask in an ice bath to ensure complete crystallization. If no crystals form, scratch the inside of the flask with a glass rod or add a seed crystal. If crystallization still doesn't occur, add a boiling chip and heat the solution in the hood to reduce the volume. After the solution has been cooled in the ice bath, filter by suction filtration, washing with several small portions of ice-cold solvent. Dry the crystals on the filter by drawing air through using continued suction or by placing in a drying oven.

Miniscale Solvent-Pair Recrystallization



This procedure should be used when a single recrystallization solvent cannot be found. Place 300 mg or more of the crude crystals in a 25-mL or larger Erlenmeyer flask. On a hot sand bath or steam bath, heat two flasks containing the solvents. One flask will contain solvent in which the compound is soluble. The other flask will contain solvent in which the compound is insoluble. Add dropwise the solvent in which the compound is soluble. Add just enough to dissolve the compound, no more. Then, add dropwise the solvent in which the compound is less soluble. Observe carefully. Add just enough until the solution turns cloudy (the cloud point) and remains so even after swirling. This is the point at which the crystals are beginning to come out of solution. Now add 1 or 2 drops (no more!) of the solvent in which the compound is soluble. Add just enough to dissipate the cloudiness. Remove the flask from the heat source and let the solution stand undisturbed to cool to room temperature. When crystallization occurs, chill the flask in an ice bath and suction filter the crystals. If crystallization does not occur, scratch the inside of the flask with a glass rod or add a seed crystal. If crystallization still does not occur, heat the solution in the hood. Again add dropwise the hot solvent that does not dissolve the compound. At the cloud point, cool the solution and wait for crystallization. After crystallization occurs, cool the flask in an ice bath and suction filter. Wash the crystals with several small portions of ice-cold solvent (the one in which the compound is insoluble). Allow the crystals to dry before taking a melting point.

If some of the solid does not dissolve during addition of the first hot solvent, filter the insoluble material by gravity filtration and continue.

Important Tips Concerning Recrystallization

1. Use an Erlenmeyer flask in the hood for recrystallization, not a beaker. The solvent can too easily boil away from a beaker or be splashed out.
2. If no precipitate forms even after the solution has been standing in an ice bath, it may be necessary to induce crystallization. Try one or more of the following techniques:
 - a. Scratch the inside surface of the Erlenmeyer flask with a glass rod. The scratched glass acts to induce crystallization.
 - b. Add a seed crystal of the compound (if available) to the cooled solution. This can act as a template to initiate crystallization.
 - c. If neither of the previous suggestions works, it is probable that too much solvent was added initially. Reduce the volume by heating until the solution appears cloudy. This is the point at which crystallization is occurring. Add a few more drops of the hot solvent until the solution is clear, then allow to cool slowly.
 - d. If crystals have still not formed, it is possible that the wrong solvent was selected, one in which the compound was too soluble. Evaporate the solvent by boiling, leaving the impure crystals. Then try again with a different solvent.
3. Experimental procedures generally do not specify the exact amount of solvent to use for recrystallization of a crude product. If a volume of solvent is specified, that amount is based on an average yield. If the mass of crystals actually obtained is significantly different from the average yield, the amount of solvent used should be scaled accordingly. Always use the minimum amount of hot solvent to dissolve the crystals.
4. Whenever an experimental procedure specifies two solvents (separated by a hyphen) to be used for recrystallization, it implies a solvent-pair recrystallization. The solvent

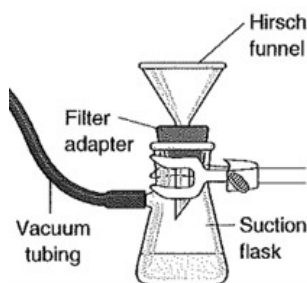
listed first is the solvent in which the compound is soluble. The second solvent is the one in which the compound is insoluble. For example, "recrystallize the solid from ethanol-water" means that the solid is soluble in ethanol and not soluble in water.

5. Oils are sometimes obtained instead of crystals. This is a common problem, especially if the solid has a low melting point. An oil can form if the boiling point of the solvent is greater than the melting point of the compound. When the oil solidifies, impurities are trapped within the crystal lattice of the crystals. If this happens, try one or more of the suggestions listed below:
 - a. Redissolve the oil by heating gently and let the solution cool back down to room temperature. If an oil starts to re-form, shake the mixture vigorously and cool in an ice bath. Repeat until solidification occurs.
 - b. Add a little more solvent to the oil. If using a solvent pair, add a little more of the solvent in which the compound is insoluble. If this doesn't work, try adding a little more of the solvent in which the compound is soluble. Let stand. If a seed crystal is available, add one now.
 - c. Triturate the oil. To triturate, add a small amount of a solvent in which the product is expected to be insoluble. With a glass rod, gently mash the oil in the solvent. If impurities in the oil are soluble in the solvent, then the oil may crystallize using this procedure.
 - d. If the above methods don't work, start over, using a new solvent for recrystallization. Use a solvent with a lower boiling point.

Introduction to Filtration

Once crystals have formed, they must be separated by the process of filtration. In filtration, a porous barrier, usually filter paper or sintered glass, allows the liquid but not the solid to pass through. The two most important methods of filtration are gravity filtration and suction (vacuum) filtration. Gravity filtration is done when the desired substance is in the solution; the solid (such as decolorizing carbon or a drying agent) is collected on the paper and discarded and the filtrate is collected for further use. Suction filtration is chosen when the desired substance is the solid and it is necessary to isolate it as a dry solid.

How to Do a Microscale Suction Filtration



This technique is used to isolate a dry solid product as in recrystallization. The equipment required is a heavy-walled, sidearm suction flask, rubber grommet ring or filter adapter (neoprene), a Hirsch funnel, a piece of appropriately size filter paper, and thick-walled vacuum tubing. The flask should be clamped to avoid breakage.

The filter paper should be large enough that the holes in the funnel are just covered. If the filter paper selected is too large (larger than the diameter of the funnel), a tight seal cannot be obtained. Additionally, some of the mixture may flow over the unsealed edge and result in loss of product as well as contamination of the filtrate. A small quantity of the solvent to be filtered is used to wet the paper, and suction is applied, usually via a water aspirator. The maximum suction should always be applied, so the aspirator is fully turned on. This procedure prevents solids from getting under the filter paper, which could lead to clogging of the funnel and/or loss of the solid into the filtrate. The mixture to be filtered is swirled and then rapidly poured into the funnel. After the liquid has been drawn through, the resulting crystals are usually washed with a minimum amount of cold solvent. If the solvent is high boiling, traces may be washed out by using a more volatile solvent, as long as it does not dissolve the filtered crystals. Drawing air through the filter for a few minutes further dries the crystals. It is important to remove all of the solvent or the yield and melting point will be inaccurate. It may be necessary to press out the last remnants of the solvent with a spatula by pressing down on the filter cake. The resulting dry, solid product is carefully removed from the filter paper, taking care not to scrape off pieces of the paper fibers and contaminate the product.

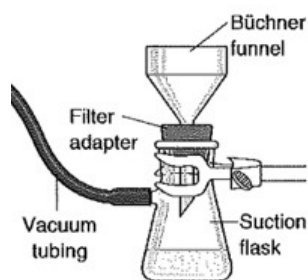
A safety trap may be used during the suction filtration to prevent backup of water into the filtrate. This can occur if there is a sudden drop in pressure, for example, if nearby aspirators are turned on. **It is also important to break the vacuum connection at the side arm of the suction flask before the aspirator vacuum is turned off or a water backup may occur.**

How to Use a Microscale Filter Pipet



Use of a filter pipet is a quick and easy method for separating a liquid from an unwanted solid, such as a drying agent. Prepare the filter pipet by placing a small piece of glass wool in the neck of a Pasteur pipet. Use an applicator stick to push it down into the tip of the pipet. Draw up the solution to be filtered into the filter pipet. Apply pressure to the pipet bulb and completely force all of the liquid out of the filter pipet into a clean flask. The drying agent or solid impurities will adhere to the glass wool. Rinse the filter pipet with fresh solvent and add this rinse to the flask. Even better, the solution may be drawn up in a clean Pasteur pipet and drained through the filter pipet.

How to Do a Miniscale Suction Filtration



The procedure for suction filtration is very similar to that of microscale suction filtration. A Büchner funnel is used in place of a Hirsch funnel. The size of the suction flask will depend upon the quantity of solution to be filtered. Because the apparatus tends to be very top-heavy, it should be clamped to a ring stand.

Place the filter paper in the funnel and pour a little of the solvent through the funnel to wet the filter paper. Turn on the aspirator or vacuum source full force to seal the filter paper to the funnel. Then carefully pour the solution in the center of the funnel. Rinse the reaction flask with a very small amount of the cold solvent for complete transfer. Rinse the crystals with a minimum amount of the cold solvent. Continue to apply vacuum to help dry the crystals. Break the vacuum, then turn off the aspirator. When the crystals are completely dry, carefully scrape the crystals from the filter paper.

Important Tips Concerning Filtration

1. Always clamp the suction filtration apparatus to a ring stand, so that the apparatus does not tip over.
2. Make sure that the filter paper is just big enough to cover the holes of the funnel. If the paper is too big, solids can get underneath and spill into the flask along with the filtrate.
3. Placing a rubber sheet over the top of the funnel and securing it with a rubber band can help dry the crystals. The suction forces the rubber sheet down on top of the crystals, helping to dry the crystals.
4. Crystals should be thoroughly dried before being carefully scraped off the filter paper. Otherwise, tiny paper fibers will contaminate the crystals.
5. With high-boiling solvents, it is best to let the crystals air dry overnight. To do this, carefully lift the filter paper and crystals out of the funnel and place on a large watch glass.