Chapter 13: Filtration

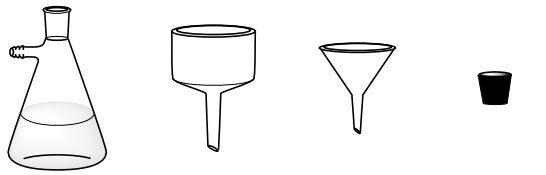
Filtration is a technique used to separate solid from liquid (the *filtrate*). The two types of filtration commonly used in organic chemistry laboratories are vacuum filtration and gravity filtration. Which method is preferred depends on whether you wish to keep the solid (for instance, a recrystallized product) or the liquid (for instance, a solution containing your product, which happens to have drying agent in it). Normally vacuum filtration is used when you wish to keep the solid and gravity filtration is used when you wish to keep the liquid, but if necessary you can use vacuum filtration for either task.

The one case where gravity filtration is absolutely necessary is when filtering a hot solution (for instance, removing insoluble impurities during a recrystallization). In this case, if you used vacuum filtration, the sudden evaporative cooling caused by air being drawn across the hot solvent might cause your dissolved compound to precipitate suddenly, clogging the funnel and making it impossible to remove insoluble particles.

Goal	Best Option
Keep solid, discard liquid	Vacuum filtration
Keep liquid, discard solid (liquid is cold)	Either vacuum filtration or gravity filtration (with cone-shaped paper)
Keep liquid, discard solid (liquid is hot and has dissolved product)	Gravity filtration (with fluted paper and stemless funnel, if available)

13.1 Vacuum Filtration

Vacuum filtration is faster than gravity filtration, because the solvent or solution and air is forced through the filter paper by the application of reduced pressure. The reduced pressure requires that they be carried out in special equipment (Figure 13-1). This includes a side-arm flask (it must be heavy-walled to withstand vacuum without imploding), a Büchner (pronounced "Byook-ner") or Hirsch funnel, and a rubber cone-shaped adapter that fits between the flask and the funnel to create an airtight seal. Büchner and Hirsch funnels are porcelain funnels with a porous support for a piece of filter paper. Büchner funnels are used for most applications in the organic chemistry teaching labs; Hirsch funnels are only needed for very small-scale reactions.



Side-arm flask | Buchner funnel | Hirsch funnel | Rubber adapter

Figure 13-1: Glassware used for vacuum filtration.

A typical vacuum filtration setup is shown in Figure 13-2.

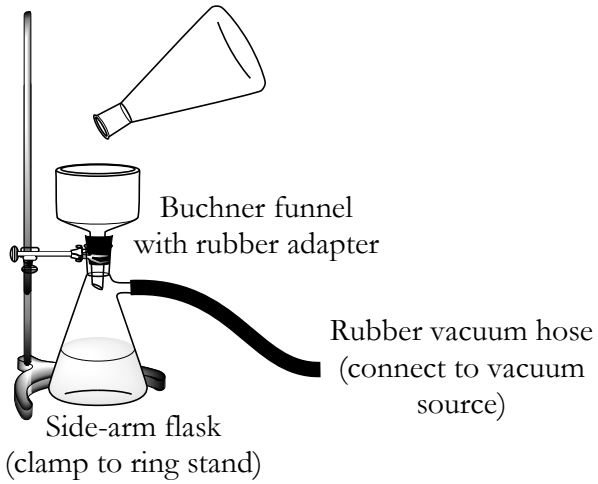


Figure 13-2: Setup for vacuum filtration.

Note that thick-walled tubing is used to attach the flask to the vacuum source because thin tubing will collapse from the low pressure. All flasks must be securely clamped to prevent them from toppling over.

The procedure for performing a vacuum filtration is as follows:

1. Assemble the apparatus.

Check the side arm flask (filtering flask) carefully for cracks, since cracks could cause the flask to break when vacuum is applied. Then, clamp the flask securely to a ring stand. Add an adapter and a funnel. Place a piece of filter paper in the funnel that is small enough to remain flat but large enough to cover all of the holes in the filter. Wet the paper with a small amount of the solvent to be used in the filtration. This causes the paper to adhere to the plate and keeps materials from passing under the paper during filtration.

2. Open the vacuum valve.

Make sure that the paper is secure on the funnel, and that air is being drawn through the paper. If you hear air whistling around the edges of the adapter, you might need to press on the funnel to engage the seal and thus the vacuum. If the filter doesn't seal well even when vacuum is applied, a small amount of water or solvent can improve the seal prior to filtering.

3. Filter the solution.

Pour the mixture to be filtered onto the filter paper. The vacuum should rapidly pull the liquid through the funnel. Watch that particulates do not creep under the edges of the paper. If this happens, start over and carefully pour portions of the solution onto the very center of the paper.

4. Rinse the solids.

Rinse the solids in the funnel (the "filter cake") with a small amount of fresh, cold solvent to help remove impurities that were dissolved in the filtrate. If you want to fully dry the solids, you can continue to draw air through them for several minutes afterwards. Note that all student hoods share the same vacuum line, so leaving the vacuum outlet open to atmosphere at your hood will reduce the

effectiveness of the vacuum system for everyone else. For this reason, it is important to close the vacuum outlet when you are finished.

13.2 Gravity Filtration

Gravity filtration is performed in either a stemmed funnel or a stemless funnel. Both are typically made of glass.

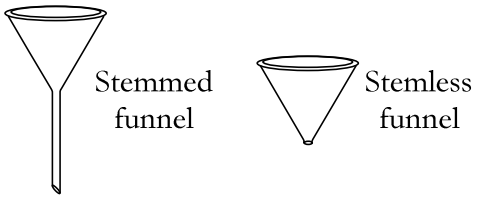


Figure 13-3: Glassware used for gravity filtration.

A. Folding Filter Paper

There are two different ways to fold filter paper for gravity filtration: either with a cone-shaped filter or a fluted filter. Cone-shaped filters are faster and easier to fold, but liquids flow through them more slowly because the entire outside face of the cone is in contact with the glass funnel. To prepare a cone-shaped filter, fold a disc of paper in half, then into quarters. Pop one of the quarters out from the other three to create a cone (Figure 13-4).

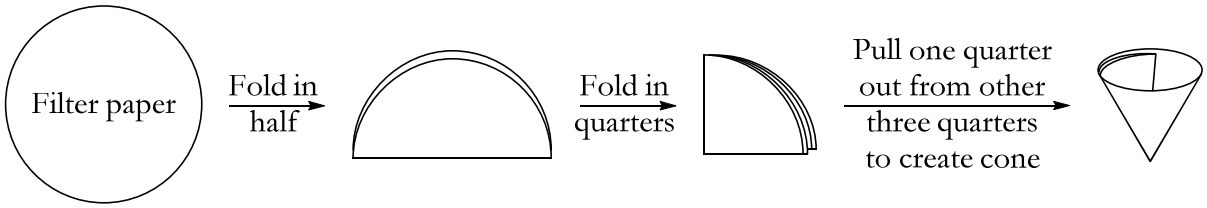


Figure 13-4: The procedure for folding a cone-shaped filter.

Fluted papers are very efficient for removing solids from organic solutions, because the fluted shape provides a larger surface for the liquid to run through and because air is able to leave the flask along the folds. Fluted papers are always used for filtering hot solutions, since they limit the contact of the hot liquid with glassware that could cool it down. To prepare a fluted filter paper, fold the paper into quarters and then crease the paper into eights, then sixteenths (Figure 13-5).

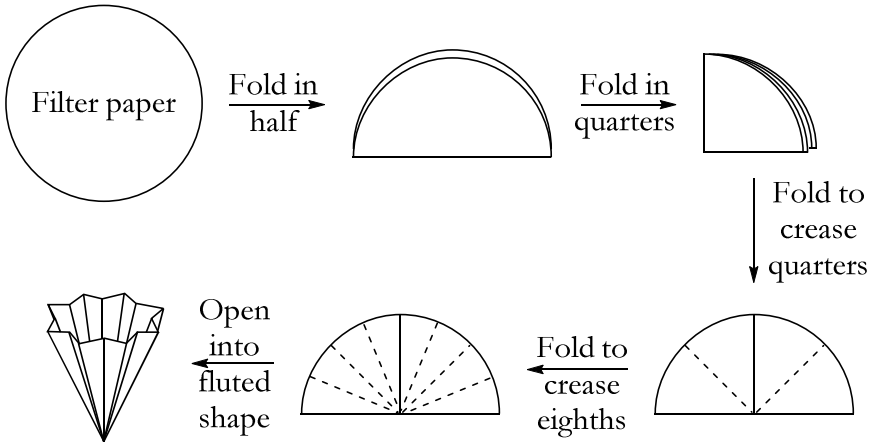


Figure 13-5: The procedure for folding a fluted filter.

B. Procedure for Gravity Filtration

1. Select and fold the filter paper.

Select the size of filter paper that, when folded, will be a few millimeters below the rim of your glass funnel. Support the glass funnel in a ring or place it in the neck of an Erlenmeyer flask. Fold it either with a cone or fluted folding.

2. Filter the solution.

Wet the filter paper with a few milliliters of the solvent to be used. Wetting the paper holds it in place against the glass funnel. Pour the mixture to be filtered through the funnel, a little at a time if necessary.

C. Procedure for Gravity Filtration of Hot Solutions

1. Select and fold the filter paper.

Fluted filter papers are always used in hot gravity filtrations.

2. Prepare the filter paper and funnel.

Place the fluted paper in a stemless funnel that is supported in a ring above a flask resting on a hotplate. A stemless funnel must be used because the compound could crystallize in the stem of a funnel due to cooling of the saturated solution. Pour a small amount of hot solvent in the funnel to wet the filter paper. Cover the funnel with a watch glass and warm the entire apparatus on the hotplate.

3. Filter the solution.

The mixture to be filtered should be warm, but not boiling. Add a 10–20% excess of solvent to allow for evaporation in the procedure. Pour the hot mixture through the funnel, in portions if necessary. Keep the entire apparatus warm and covered at all times. If crystals begin to form at any time during the process, add a small portion of warm solvent to dissolve them.

13.3 Other Filtration Methods

In rare cases you might need to use a specialized filter of some other kind.

A. Celite

Some precipitates are so fine that they pass right through filter paper. The filtration capabilities of the paper can be increased by coating it with a layer of finely ground, inert diatomaceous earth called Celite. This method is only used when the precipitate being collected is not the desired product, since it would be difficult to separate out the Celite from the solid afterwards. The procedure for doing this is the same as for vacuum filtration, but before filtering your product you should fill the Buchner funnel about halfway with Celite and then wet it down with solvent.

B. Filtering Pipets

Filtering pipets are used to filter solid particles from a very small amount of organic solvent. A filtering pipet is simply a Pasteur pipet with a small plug of cotton or glass wool inserted snugly at the beginning of the lower constriction in the pipet. Use another Pasteur pipet to add the liquid to the top of the filter pipet. If you want to push the liquid through the filter more quickly, you can add a pipet bulb to the top of the pipet to force it through.

C. Syringe Filters

Syringe filters (Figure 13-6) are designed to attach to the bottom of a syringe. They are commercially available in several different materials (nylon and cellulose are the most common) and pore sizes (0.2 μm and 0.45 μm are the most common). The filter itself is encased in a small, round plastic casing with threads that screw onto the syringe. These filters are useful for filtering very small volumes of liquid, since almost none of the liquid is absorbed into the filter or lost to evaporation. They are also a good choice for removing very fine particles that would not be trapped by filter paper.

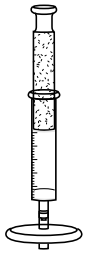


Figure 13-6: A syringe with a syringe filter attached.