• FILTRATION METHODS

There are several filtration methods: simple or gravity, hot and vacuum filtrations. The selection of the appropriate method is typically dictated by the nature of the experimental situation. The answers to each these questions help dictate what type of set up is required.

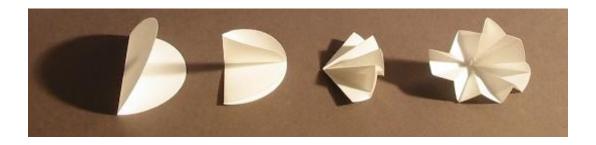
- 1. Are you collecting the solid or the filtrate (solution)? If you are collecting the solid, vacuum methods are likely best as they are faster. If you are collecting the filtrate (solution), gravity filtration methods are often preferred.
- 2. How much solution is there to filter? If you have a large volume to filter, gravity filtration is probably the better choice. Vacuum filtration is fast but if you need to empty the flask part way through you have to release the pressure and remove the funnel to do this which increases the risk of spills and slows things down.
- 3. How much solid is being collected? If only a small amount is being collected, a vacuum filtration using the Hirsch funnel is preferred.
- 4. How fine is the solid? If it's a fine solid, regular filtration might be very slow, so use vacuum filtration.
- 5. Will unwanted crystallisation occur in the filter funnel? If yes, use hot filtration.

a. The filter paper

First, make sure you use the appropriate size filter paper. This is most important for vacuum filtration where the paper should smaller in diameter than the base of Buchner or Hirsch funnel (but it must cover all the holes) and should sit flat on the bottom of the funnel with no creases or folds.

Second, folding the filter paper. There are two ways to fold filter papers, the "conventional" method and "fluted".

Here are two videos showing how to fold filter papers. (version 1, version 2)



The steps to flute the filter paper are shown. First, fold in half; open and fold in half at 90° to the first fold, subsequently align adjacent folds and make new folds bisecting the previous folds until a fan-like arrangement is obtained. Pleat into a fan by folding each segment in the opposite direction to its neighbours, in accordion-like fashion. When opened out the complete fan-like fluted paper results.

Fluting the filter paper maximises the rate at which the liquid may flow through the filter paper by increasing the surface area and by allowing air to enter the flask along its sides to permit rapid pressure equalisation.



b. Gravity or simple filtration

This is the most common method of filtration and is used to remove an insoluble solid material from a solution. The solid could be the required product or an impurity or an additive such as a drying agent. A filter paper is folded (conventional or fluted) and placed in a filter funnel which is then placed in the neck of an Erlenmeyer flask or supported in a clamp or ring stand. The solution to be filtered is then slowly and carefully poured into the funnel taking care not to fill the funnel above the edge of the filter paper. Here are two videos showing this filtration method (version 1, version 2)



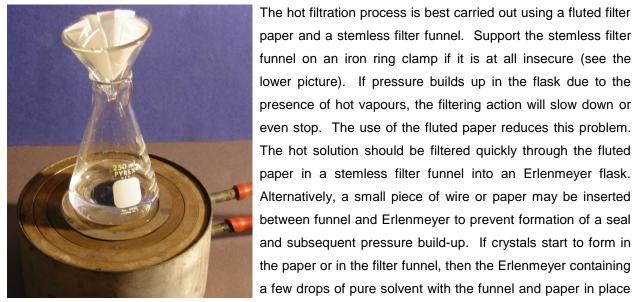
Gravity filtration with a fluted filter paper

c. Hot filtration



Never heat organic solvents with a Bunsen burner. Use a hot plate or a hot water bath on a hot plate.

Sometimes during a gravity filtration, crystals can start to grow in the filter funnel and may block the funnel, stopping filtration. This problem can be avoided by using a hot filtration where the whole filtration apparatus is heated in order to prevent the solution from cooling significantly.



paper and a stemless filter funnel. Support the stemless filter funnel on an iron ring clamp if it is at all insecure (see the lower picture). If pressure builds up in the flask due to the presence of hot vapours, the filtering action will slow down or even stop. The use of the fluted paper reduces this problem. The hot solution should be filtered quickly through the fluted paper in a stemless filter funnel into an Erlenmeyer flask. Alternatively, a small piece of wire or paper may be inserted between funnel and Erlenmeyer to prevent formation of a seal and subsequent pressure build-up. If crystals start to form in the paper or in the filter funnel, then the Erlenmeyer containing a few drops of pure solvent with the funnel and paper in place should be heated on a steam bath (or a hot plate) and the entire filtration procedure carried out on the steam bath. Hot solvent may be added to remove any crystals appearing in the filter paper.



Do not use metal tongs or a test-tube holder to hold the hot Erlenmeyer flask while pouring the liquid since it is too easy to drop the flask. A better convenient simple holder may be fashioned from a piece of paper towel folded into a strip and pinched around the neck of the flask.

Here is a video showing the hot filtration method.

d. Vacuum filtration



Vacuum filtration with a Buchner funnel

In a vacuum filtration, the solution to be filtered is drawn through the filter paper by applying a vacuum to a filter flask with a side arm adaptor (also known as a Buchner flask). Vacuum filtration is typically a fast and efficient way of filtering.

The crystals are collected by swirling the mixture of the solid and liquid and then pouring quickly it into the filtration apparatus. This typically comprises a Büchner funnel fitted with the appropriate size filter paper; a <u>clamped</u> filter flask with conical filter adapter, and a vacuum applied to the side arm of the filter flask (see left).

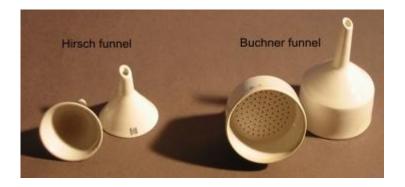


Vacuum filtration with a Hirsch funnel

If smaller quantities are to be filtered the Hirsch funnel (see left and below) and a small filter paper should be used instead. If needed, the filter flask can be replaced by a test tube with side arm. Again, the tube should be clamped and the vacuum applied at the side arm using the thick walled vacuum tubing.

When using vacuum filtration, it is **very important** that the correct size of filter paper be used. The filter paper should be flat (*i.e.* not folded up at the edges) and should cover **all** the holes in the base of the funnel. It is also important that the apparatus be clamped since it is very easily tipped over usually resulting in loss of the sample.





To filter the crystals, assemble the apparatus, ensuring that the funnel is sealed into the flask; turn the aspirator tap full on; moisten the filter paper with a little cold solvent to adhere the paper to the funnel, to prevent crystals from creeping under the paper and through the filter. The mother liquor containing the crystals may now be filtered. The crystals should then be rinsed with a small portion of cold solvent. Allow the crystals to remain exposed to the vacuum for a few minutes in order to dry them. DISCONNECT THE TUBING BEFORE TURNING OFF THE ASPIRATOR TAP. Thick-walled pressure tube is used throughout the apparatus. A trap is used in conjunction with the water aspirator. If the water pressure in the laboratory line drops suddenly, the pressure in the filter flask may become less than that in the water aspirator. This would cause water to be drawn from the aspirator stream into the filter flask and would contaminate the filtrate. The trap stops this reverse flow. A similar reverse flow would occur if the water flow at the aspirator were stopped before disconnecting the tubing to the aspirator side-arm or opening the valve on the top of the water trap.