Suction Filtration Overview

Suction filtration (vacuum filtration) is the standard technique used for separating a solid-liquid mixture when the goal is to retain the solid (for example in crystallization). Similar to gravity filtration, a solid-liquid mixture is poured onto a filter paper, with the main difference being that the process is aided by suction beneath the funnel (Figures 1.70 + 1.71).

Figure 1.70: Suction filtration.

The process has advantages and disadvantages in comparison to gravity filtration.

Advantages: 1) Suction filtration is much faster than gravity filtration, often taking less than one minute with good seals and a good vacuum source. 2) Suction filtration is more efficient at removing residual liquid, leading to a purer solid. This is especially important in crystallization, as the liquid may contain soluble impurities which could adsorb back onto the solid surface when the solvent evaporates.

Disadvantages: The force of suction may draw fine crystals through the filter paper pores, leading to a quantity of material that cannot be recovered from the filter paper, and possibly an additional quantity that is lost in the filtrate. This method therefore works best with large crystals. On small scales, the loss of material to the filter paper and filtrate is significant, and so other methods are recommended for microscale work.
Rinsing

As the goal of suction filtration is to fully separate a solid from its surrounding liquid, rinsing the solid is necessary if the liquid cannot easily evaporate. In the case of crystallization, the liquid may contain impurities that can reincorporate into the solid if not removed.

To rinse a suction-filtered solid, the vacuum is removed and a small portion of cold solvent is poured over the solid (the "filter cake"). In the case of crystallization, the same solvent from the crystallization is used. The solid is then delicately slushed around in the solvent with a glass rod, and the vacuum is reapplied to remove the rinse solvent.

To demonstrate the importance of a rinse, Figure 1.72 shows the recovery of a white solid from a yellow liquid using suction filtration. The yellow liquid seemed to be somewhat retained by the solid, as the first crystals collected had a yellow tint (Figure 1.72b). However, rinsing with a few portions of cold solvent were effective at removing the yellow liquid (Figure 1.72d), which could have been reincorporated into the solid without the rinse.

Water Aspirator

A vacuum source is necessary for suction filtration (and vacuum distillation). Although many science buildings come equipped with a house vacuum system (Figure 1.73a), solvents evaporating from a suction filter flask over time can degrade the oil pumps used in a house vacuum. Therefore, it is recommended to instead connect a suction flask to a water aspirator.

A water aspirator is an inexpensive attachment to a water spigot, and the nub on the aspirator connects with tubing to the vessel to be evacuated (Figure 1.73b). As water flows through the faucet and the aspirator, suction is created in the flask.
A water aspirator creates suction through the Bernoulli Principle (technically, the Venturi Effect, for liquids). Water coming from the faucet is constricted inside the aspirator (Figure 1.73c). As the water flow must be the same going into the aspirator as it is going out, the water speed must increase in the constricted area in the direction of flow. A similar phenomenon can be seen in creeks and rivers where the water flows the fastest at the narrowest portions of streams. When the water increases its velocity in the direction of the water flow, conservation of energy dictates that its velocity in perpendicular directions must decrease. The result is a lowered pressure adjacent to the fast-moving liquid. In other words, the gain in velocity of the constricted liquid is balanced by a reduction in pressure on the surrounding material (the gas).

For this reason, the speed at which the water flows through the faucet is correlated with the amount of suction experienced in the connected flask. A strong flow of water will have the fastest speeds through the aspirator and the greatest reduction in pressure.

Step-by-Step Procedures

Assemble the suction filtration flask

1. Clamp a side-arm Erlenmeyer flask to a ring stand or latticework and attach a thick-walled rubber hose to its side arm. Connect this thick tubing to a "vacuum trap" (Figure 1.74) and then to the water aspirator. It is best to not bend or strain the tubing as much as is practical, as this may cause poor suction.

A vacuum trap is necessary when connecting apparatuses to a vacuum source as changes in pressure can cause back-suction. When using a water aspirator, back-suction might cause water from the sink to be pulled into the vacuum line and flask (ruining the filtrate), or the filtrate to be pulled into the water stream (contaminating the water...
supply).

Figure 1.75: a) Placing the Buchner funnel in a rubber sleeve and Erlenmeyer flask, b) Concavity of a filter paper, c) Placing filter paper in a Buchner funnel, d) Placing filter paper in a Hirsch funnel.

2. Place a rubber sleeve (or filter adapter) and Buchner funnel atop the side-arm Erlenmeyer flask (Figure 1.75a). Alternatively use a Hirsch funnel for small scales (Figure 1.75d).

3. Obtain a filter paper that will fit perfectly into the Buchner or Hirsch funnel. Filter papers are not completely flat and have a subtle arc to their shape (Figure 1.75b). Place the filter paper inside the funnel concave side down (Figure 1.75b+c). The paper should cover all the holes in the funnel, and with the paper arcing downward (Figure 1.76a), solid will be less likely to creep around the edges.

Figure 1.76: a) Filter paper in the funnel, b) Wetting the filter paper with solvent, c) Pressing the Buchner funnel to create a good seal, d) Testing the suction of the aspirator.

4. Turn on the faucet connected to the water aspirator to create a strong flow of water (the degree of suction is related to the water flow). Wet the filter paper with cold solvent (using the same solvent used in crystallization, if applicable, Figure 1.76b).

5. Suction should drain the liquid and hold the moist filter paper snugly over the holes in the filter. If the solvent does not drain or suction is not occurring, you may need to press down on the funnel (Figure 1.76c) to create a good seal between the glass and rubber sleeve.

Lack of suction may also be from a faulty aspirator or a leak in the system: to test for suction remove the tubing from the suction flask and place your finger over the end (Figure 1.76d).

Figure 1.77: a) Using a spatula to dislodge a thick solid from the glass, b) Filtering, c) Using a spatula to scoop a thick solid onto the filter paper, d) Rinsing residual solid from the flask using cold solvent.

Filter and Rinse the Mixture

6. Swirl the mixture to be filtered in order to dislodge solid from the sides of the flask. If the solid is very thick, use a spatula or stirring rod to free it from the glass (Figure 1.77a).
In the context of crystallization, the flask will have previously been in an ice bath. Use a paper towel to dry water residue from the outside of the flask so water does not accidentally pour onto the solid.

7. With a quick motion, swirl and dump the solid into the funnel in portions (Figure 1.77b). If the solid is very thick, scoop it out of the flask onto the filter paper (Figure 1.77c).

It's best if the solid can be directed toward the middle of the filter paper, as solid near the edges may creep around the filter paper.

8. A small amount of chilled solvent (1-2 mL for macroscale work) can be used to help rinse any residual solid from the flask into the funnel (Figure 1.77d). In crystallization, it is not wise to use an excessive amount of solvent as it will decrease the yield by dissolving small amounts of crystals. Again, press on the funnel to create a good seal and efficient drainage if necessary.

9. Rinse the solid on the filter paper to remove contaminants that may remain in the residual liquid.
   a. Break the vacuum on the flask by opening the pinch clamp at the vacuum trap (Figure 1.78a) or by removing the rubber tubing on the filter flask. If adjusting the pinch clamp, you will know the system is open when there is an increase in water flow by the faucet. Then turn off the water on the aspirator. It is always important to open the system to the atmosphere before turning off the aspirator in order to prevent back-suction.
   b. Add 1-2 mL of cold solvent (Figure 1.78b). Use a glass stirring rod to break up any solid chunks and distributed the solvent to all portions of the solid (Figure 1.78c), taking care to not rip or dislodge the filter paper.
   c. Reapply the vacuum to the flask, and dry the solid with suction for a few minutes.

10. After filtration is complete, again open the flask to the atmosphere by releasing the pinch clamp or opening it elsewhere, and turn off the water connected to the aspirator.

11. Transfer the solid, filter paper and all, to a pre-weighed watch glass using a spatula (Figures 1.79a+b). The filter cake shouldn't be mushy, and if it is, the liquid was not adequately removed (try a different aspirator and repeat suction filtration).

12. Allow the solid to dry overnight if possible before recording a final mass or melting point. The solid will flake off the filter paper more easily when completely dry (Figure 1.79c).
13. If pressed for time, a solid can be quickly dried in the following ways:

- If the solid is wet with water, it can be placed in a \(110^\circ C\) oven (if the melting point is not below this temperature). If the solid is wet with organic solvent, it should never be placed in an oven as it may ignite.
- If the solid is wet with organic solvent, it can be pressed between fresh pieces of filter paper (multiple times if needed) to quickly dry them. Inevitably some solid will be lost on the filter paper.

**Suction Filtration Summary**

![Figure 1.80: Suction filtration vacuum trap apparatus.](image)

Clamp a side-armed Erlenmeyer flask.

Connect thick-walled hosing from the side arm to a vacuum trap and the water aspirator.

Place a vacuum sleeve on the Buchner (or Hirsch) funnel, then filter paper on.

Turn on the aspirator.

Add a few \(\text{mL}\) of the same solvent used in the flask to wet the filter. The solvent should drain with suction.

Swirl the mixture to be filtered to dislodge the solid from the sides of the

In some applications, (e.g. crystallization), rinse with solvent:

- Open the apparatus to the atmosphere, then turn off the aspirator.
- Add a few \(\text{mL}\) of cold solvent to the filter

Apply suction again for a few minutes (repeat the rinse step if necessary).

Dry the solid on a watch glass along with the filter paper, overnight if possible. The solid will flake off the paper when dried.
the funnel so it arches downward. With a quick motion, pour the slurry into the funnel in portions.

• Delicately swirl the solid in the solvent with a glass rod.

Table 1.10: Procedural summary for suction filtration.

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