

## Separatory Funnel Extraction Procedure

(Adapted from on-line instructions from the University of Colorado at Boulder:  
<http://orgchem.colorado.edu/hndbksupport/ext/extprocedure.html>)

### 1. Inspect your separatory funnel.

There exist many different styles of separatory funnels, with different types of stopcocks and stoppers, as illustrated in the photo below. Generally, the Teflon stopcocks work better than the ground glass stopcock. However, the Teflon-style stopcocks may leak if they are not properly assembled.



*The sep funnel on the left is a 60 mL size, the others are 125 mL.*

There are two different styles of stopper, too. Some students swear by the plastic style, some by the ground glass style. The disadvantage of the ground glass style is that it can lodge permanently in the sep funnel if it is not removed and stored separately after use. Whichever style you have, make sure that the stopper fits snugly in the top of the flask.



**2. Support the separatory funnel in a ring on a ringstand.**

You will be provided with a ring stand and a ring. Stands and rings can come in many different sizes. You should check to be sure that the ring you have chosen is compatible with the separatory funnel that you are using. Some people like to add pieces of cut tubing to the ring in order to cushion the funnel.



*Make sure the stopcock of the separatory funnel is closed!*

**3. Add the liquid to the separatory funnel.**

Place a stemmed funnel in the neck of the separatory funnel. Add the liquid to be extracted, then add the extraction solvent. If you are using a 125-mL funnel, then the total liquid volume in the separatory funnel should probably be around 100 mL. After pouring in the liquids, insert the stopper in the neck of the separatory funnel.



*pour in the liquid to be extracted...*



*pour in the solvent...*



*add a stopper*

#### 4. Shake the separatory funnel.

Pick up the separatory funnel with the stopper in place and the stopcock closed, and rock it once gently. Then, point the stem up and slowly open the stopcock to release excess pressure. Close the stopcock. Repeat this procedure until only a small amount of pressure is released when it is vented.



*rock gently... don't shake vigorously (yet)*



*point the stem up and away from you when you open the stopcock to release pressure*

Now, shake the funnel vigorously for a few seconds. Release the pressure, then again shake vigorously. About 30 sec total vigorous shaking is usually sufficient to allow solutes to come to equilibrium between the two solvents.

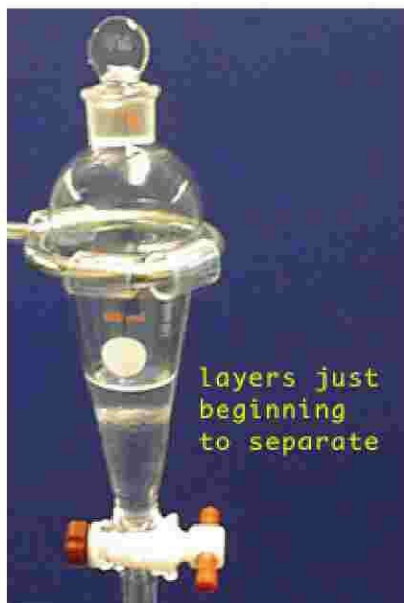


*shake vigorously once you are sure there is no pressure build-up*



Vent frequently to prevent pressure buildup, which could cause the stopcock (and perhaps hazardous chemicals) to blow out. Take special care when washing acidic solutions with bicarbonate or carbonate since this produces a large volume of CO<sub>2</sub> gas.

**5. Separate the layers.**



*1. Let the funnel rest undisturbed until the layers are clearly separated.*



*2. While waiting, place a beaker under the funnel. If the solvent is not too volatile, you can remove the stopper.*



*3. After the layers have separated completely, be sure the stopper is out; then open the stopcock and allow the lower layer to drain into the flask.*



*4. If the upper layer is to be collected from the funnel, remove it by pouring it out of the top of the funnel.*

**6. Perform multiple extractions as necessary.**

Often you will need to do repeat extractions with fresh solvent. You can leave the upper layer in the separatory funnel if this layer contains the compound of interest. If the compound of interest is in the lower layer, the upper layer must be removed from the separatory funnel and replaced with the drained-off lower layer, to which fresh solvent is then added.

Sometimes, beginning students do not know in which layer resides the compound of interest. The best advice: Always save all layers until the experiment is completely finished!

**7. Store your separatory funnel with the cap (stopper) separate from the funnel!**



If you do not store the cap separately, it can become irreversibly attached to the sep funnel. This is a common problem in teaching labs: a student finds a cap lodged in the sep funnel. Yes, there are ways to remove the stopper...sometimes. In many the cases, while trying to remove a stubborn stopper, the separatory funnel is broken. Separatory funnels cost about \$50 each. So, simply because a student doesn't remove the stopper before storage, a \$50 piece of glassware is ruined.

Please, remove the stopper before storage!