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## 7.8. Distillation Guide

### Overview:

Distillation is an extremely useful technique that is used to purify reagents and separate crude product mixtures. There are two varieties of distillation - atmospheric pressure and reduced pressure. The former is easier, while the latter involves some more complicated techniques. Both types will be explored in 5.301.

### Glassware:

Distillations require special glassware that is unique to this technique. There are several types of set-ups, but we will use only two. In both cases we will use a short path distillation head, varying only in the use of a Vigreux column. Even though we won't use other set-ups in 5.301, you should become familiar with them through your reading.

### Steps for Running an Atmospheric Pressure Distillation:

- 1) Collect the necessary glassware:** short path distillation head, thermometer and adapter, receiving flasks (at least two), Vigreux column (optional - consult LLP page 196).
- 2) Preheat oil bath or heating mantle.** If the boiling point is unknown, this step should be omitted. Keep in mind that for most distillations the heating apparatus must be 20–30 °C higher than the boiling point of the distillate. *Note: Due to thermal breakdown and possible ignition, oil baths are only useful for temperatures below 200 °C.*
- 3) Record weight of labeled receiving flasks.**
- 4) Put compound to be distilled in a round-bottomed flask with stir bar.** (The stir bar will prevent bumping.) The size of the round bottom flask is very important. It should be roughly half to two-thirds full; any higher and it may boil over prematurely, any less and it may take too long to distill.
- 5) Assemble glassware, making sure all joints are snug.** A large assembly of glassware should involve at least two clamps - when in doubt use more clamps! Also, no joint grease is necessary for atmospheric pressure distillations. (Note: for air or water sensitive compounds the apparatus should be flame dried and distilled under N<sub>2</sub> or Ar. We won't do this in 5.301, but you may encounter this in your UROP.)

**6) Insulate the column.** When using a Vigreux column, it should be wrapped with glass wool and aluminum foil. Without insulation, these set-ups tend to take a very long time.

**7) Connect the condenser to the water lines, turn on the water, and check for leaks.**

**8) Raise the stir plate and heating apparatus to the flask and begin heating.** Note: Variac gauges do not correspond to temperature. Setting the dial at 70 will not heat your oil bath to 70 °C - it will actually go much higher. Also, different oil baths and heating mantles will heat to different temperatures at the same Variac settings.

**9) Lower the hood sash.** This is always a good practice in case of accident, but it also keeps the distillation apparatus away from the air conditioning of the lab. This will cool your set-up and make your distillation take longer.

**10) DO NOT HEAT TOO QUICKLY!!!** Patience is the key to distillation.

**11) Slowly increase the temperature of the heating apparatus until the solution is refluxing.**

**12) Wait to see the distillation thermometer respond.** If nothing happens after about 10 minutes then raise the temperature *slightly*.

**13) Repeat step #12 until you see the distillation thermometer respond.** Once this happens, prepare to collect.

**14) Try to keep the apparatus at a constant temperature** - at least within 5 degrees of the temperature when the distillation thermometer registered.

**15) Collect until a dramatic change in temperature occurs.** Usually the temperature of the distillation thermometer will drop when one fraction is done distilling. At this point you should change receiver flasks or stop the distillation entirely.

**16) When you have collected everything you want, drop the heating apparatus and let the entire apparatus cool.**

**17) Weigh the collection flask(s) and obtain the weight of your product(s).**

### **Steps for a Reduced Pressure Distillation:**

**1) Collect the glassware** - the same as above except this time make sure to include a pig (3-neck) or cow (4-neck) adapter. Another useful piece of glassware that we won't use in 5.301 is a Perkin Triangle. This is described in your texts and may be useful later in your chemistry career.

**2) Perform steps 2–4 above.**

**3) Assemble the glassware, making sure to grease all of the joints.** Be sparing with the vacuum grease - it's expensive and you don't want it getting into your compound. See Zubrick pages 53-55 for a discussion of joint greasing.

**4) Perform steps 6–7 above.**

**5) DO NOT START HEATING!!!**

**6) SLOWLY open the distillation apparatus to vacuum.** You should see the liquid begin to bubble. Don't worry, this is normal. Excess solvent or low boiling impurities will often boil away under vacuum at room temperature. (This is a good example of why you need to keep your trap full of liquid nitrogen, otherwise these compounds will go directly into your pump oil!)

**7) Once the bubbling subsides, or slows almost to a stop, then you can start heating the flask.**

**8) Perform steps 9–15 above.**

**9) Release the vacuum.** When you are done collecting, it is not quite time to cool the apparatus. First, you must release the vacuum. Before you do this, however, make sure that all of your collection flasks are secured to the apparatus by clamps, joint clips, your hand, etc. You do not want to release the vacuum then see your product flask shatter on the

bottom of the hood! Once everything is secure, vent the apparatus to nitrogen and then remove the heating apparatus and let the set-up cool to room temperature

**10) Once everything has cooled, record the weight of your tared collection flask(s) and calculate the weight of your product(s).**