Simple Distillation of Coffee

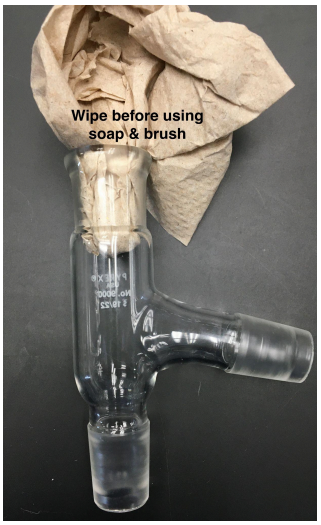
Background

Distillation is a purification technique in which compounds with different boiling points are separated. In this lab you will perform a simple distillation of aqueous mixtures including coffee.. Simple distillation is used to separate two or more compounds that have at least a 40-50°C difference in boiling point. Coffee extract contains hundreds of known chemicals. Volatile (low-boiling) compounds create the aroma; other compounds, including caffeine, are responsible for the bitter taste.

Jointware

Before discussing the distillation setup, it is necessary to learn about the jointware used in it. Ground glass joints offer a quick and easy way to build various air-tight apparatus used in organic chemistry labs. All glass joints used in this lab are standard taper size 19/22; they are made to be interchangeable such that any apparatus can be assembled from basic pieces. Standard joints are convenient but costly to manufacture. They account for much of the cost in your kit.

Ground glass joints have a rough surface and when organic compounds solidify inside the joints it occasionally can cause them to "freeze" or get stuck, sometimes permanently which renders the two pieces useless. To prevent freezing, put a small dot of grease on the top of the inner joint, then rotate the two pieces until you see a clear band going around the top part of the joint.



<u>*Please note: grease can get into the products you have just purified and may be very difficult to remove.*</u> <u>Therefore:</u>

a) Only grease joints that get hot or are on flasks storing solids (in this class you only need to grease still pot joints and glass stoppers). Don't grease other joints as other joints don't tend to stick.
b) Add your reagents to the still pot <u>*before*</u> you grease so that they are not contaminated.
c) Only grease the top of your joint, grease on bottom is likely to drip into your reaction flask. If the entire joint is clear, you used too much, remove all the grease with a paper towel and start again.
d) At the end of the experiment, wipe off grease with a paper towel <u>*before*</u> washing. Glassware with a cloudy appearance usually means someone forced grease into the flask with a wash brush.

Clamping and Clipping Jointware

The general rules of clamping are to *use a minimum number of clamps* and *build from the bottom up*.

- The most important clamp in a glassware apparatus is the lowest one, in this case, the one on the still pot. This is the first clamp to go on and it is the only one that should be tight.
- Once you have the lowest piece of your setup secured with a clamp, let each new piece settle in by gravity.
- Use additional clamps sparingly – they should be snug, but not tightened with force. Over tightening a supporting clamp may cause that piece of glassware to move.
    - Sometimes, as you tighten, the once snug joint will be lifted slightly and you will lose your airtight seal.
    - Even worse, tightening a clamp too much could force the two pieces together unnaturally and the joint could crack.

Build from the bottom up and allow joints to settle naturally! Don't over-clamp or force joints!

Aside from the standard metal clamps, we will also be using blue plastic Keck clips to hold joints together. Keck clips have a wide semicircle and a narrow semicircle. The wide end should go around the larger (female) end of the joint. Keck clips can melt, so they are never placed on joints near the heat source.

Students break clips by:

- Forcing them on upside down with the wide end on the narrow side of the joint.
- Putting them on one side of the joint instead of straddling the two pieces
- Melting them on a still pot or joint that gets hot.

Before you use a Keck clip, gently tug open the ends to check for cracks in the plastic. Broken clips are ineffective and will result in broken glassware – bring your instructor any clip that appears to be broken.

Setting up a Simple Distillation

The heat source used in most of the distillations in the class will be a blue heating mantle which is made to accommodate 50-500mL round bottom flasks. No matter how you heat, you must have a way to quickly remove the heat source if something goes wrong. (ex. you forgot to add something to the reaction; the reaction catches on fire.) The mantle should be on a partially extended lab jack to allow lowering in case of an emergency.

The round bottom flask that contains the liquid to be distilled is called the still pot. The still pot is always the first piece of glassware to be assembled. It should be tightly clamped to a ring stand. The flask should be fully lowered in the heating mantle to get the best contact with the heat source.

When heat is applied to the system, the liquid in the still pot absorbs heat until it reaches its boiling point. As the liquid is changed to vapor it travels from the still pot to the still head (a.k.a. three-way connector). The still head connects the still pot to the condenser and is the area where the thermometer is placed to read the vapor temperature. No clamps should be used on the still head and no Keck clips should be used where the still head meets the still pot.

You have two jacketed tubes in your kit; the one with the narrow inner tube is the condenser. Attach water hoses to the inlet and outlet before you assemble the condenser. (Attaching hoses later puts excess pressure on the apparatus.) Bring the condenser to the still head and don't let go. Figure out approximately where you need a supporting clamp on the condenser. A Keck clip can help you keep the joint in place while you clamp the condenser but continue to support it with your hands until it is clamped. Remember only the still pot clamp should be very tight. Attach the lower hose to the water outlet and put the end of the higher hose in the sink. This ensures that the cooling jacket will entirely fill. Slowly turn on the water. As long water is dripping into the sink, there is enough flow to keep the tube cold. Fast flowing water is wasteful and increases the chances of a leak. Check periodically during the experiment that water is still flowing. Make sure that the hoses don't come near the mantle or they will melt.

Attach a vacuum adapter to the end of the condenser with a Keck clip. If you forget the Keck clip, the joint will eventually slip off and you will have broken an expensive piece of glassware.

The receiving flask is usually a small round bottom flask that can be attached to the vacuum adapter using a Keck clip. The flask should be given extra support with a ring clamp or a three-prong clamp. Products may also be collected into vials or graduated cylinders depending on the experiment.

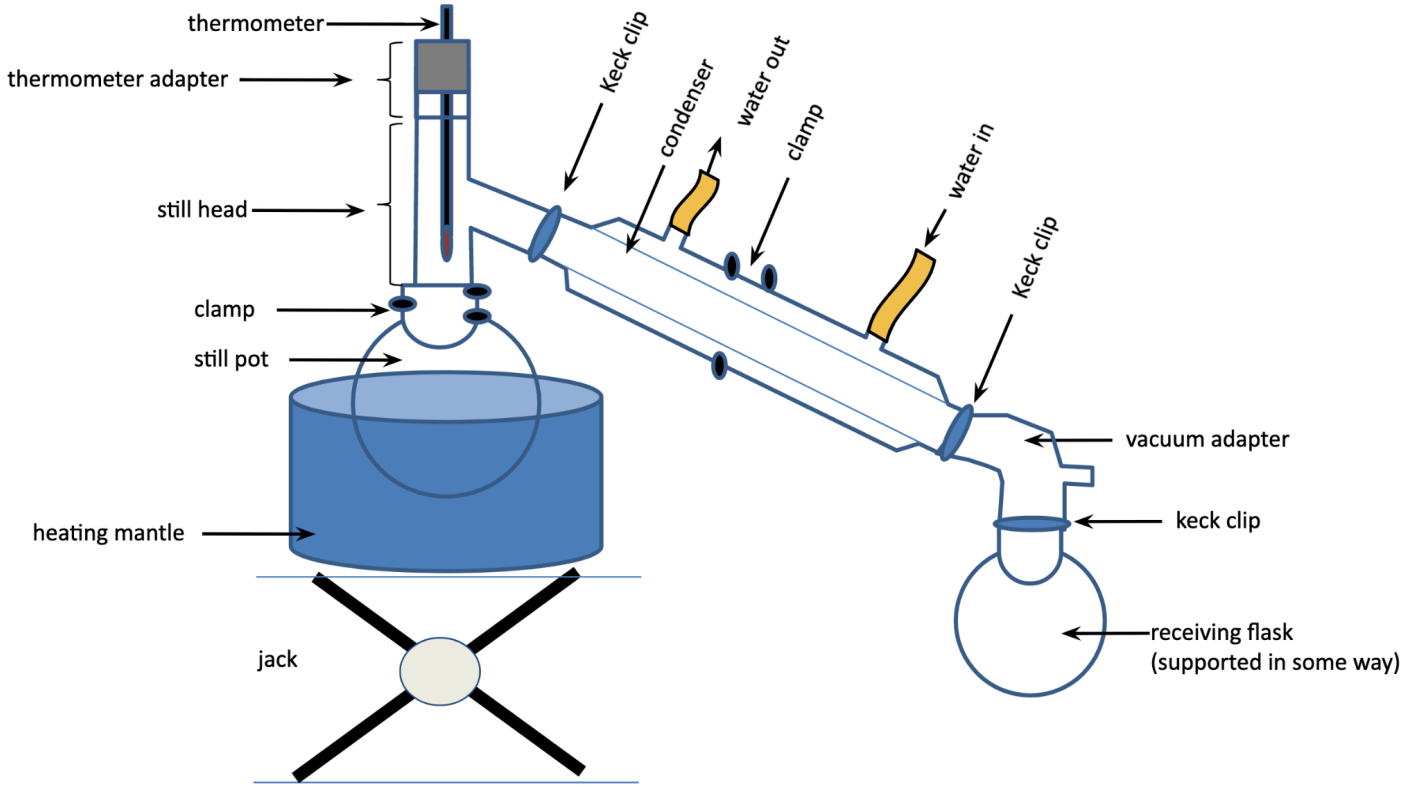
The last piece to be assembled is the thermometer adapter. The adapter has a glass part and a rubber part that fit together. The ground joint of the glass piece fits the top of the still head. The thermometer fits through a hole in the rubber piece. Put these two pieces together, if they are not already, and push the thermometer through a few inches beyond the glass joint. Place the adapter on top of the still head and note the position of the thermometer. To read the distillation temperature correctly, the bottom of the probe should be below the side-arm leading to the condenser. (See image on the right.) Do not adjust the thermometer while it is on the apparatus. Remove the adapter, push the thermometer, replace the adapter and repeat until you have the correct placement.

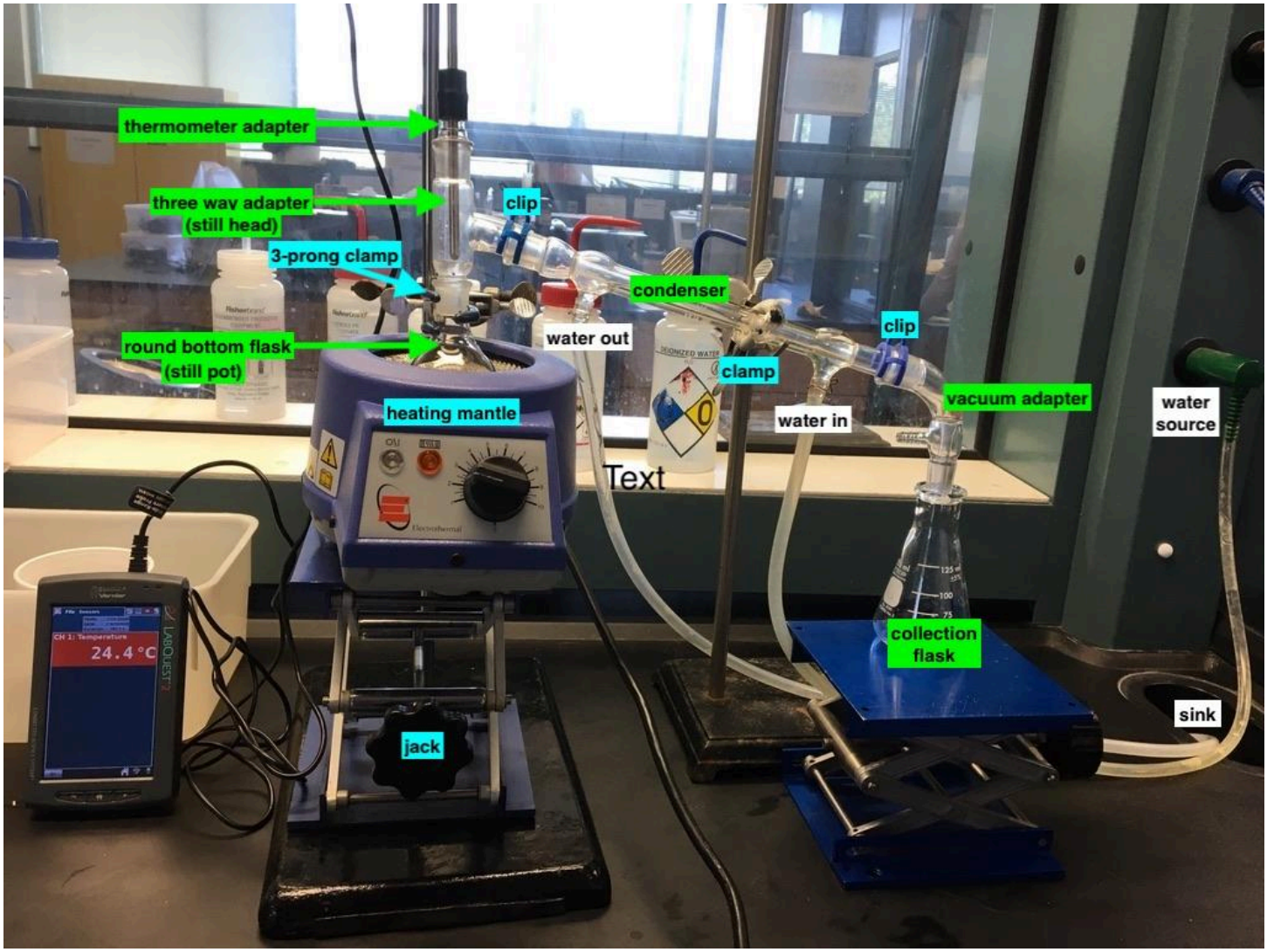
Distillation Theory

For simple distillation, the compounds to be separated must have at least a 40°C difference in boiling point. When the contents of the still pot are heated, initially, only the more volatile component becomes a vapor and starts rising. Less volatile components remain liquid in the still pot. When the vapors begin condensing on the bulb of the thermometer, the temperature reading rises until it reaches the boiling point of the liquid being distilled. As vapor travels down the condenser it hits the cool glass, condenses back into a liquid and drips into the receiving flask.

The temperature range where you start and stop collecting liquid in the receiver should be recorded as the boiling point. Like melting points, boiling points are really a range. During a simple distillation, if the temperature stays relatively constant, it is likely that you are distilling one product. If the temperature starts dropping sharply, the most volatile compound has finished distilling and the second component is still heating, not yet vaporizing. If the temperature starts rising sharply, the most volatile component has finished distilling and the second, higher boiling, compound is starting to distill. In either case, you should stop collecting your product.

Never distill the still pot to dryness in this or any distillation. Chemicals remaining in the still pot may cause an explosion or fire. The mantle will give off residual heat even after it is turned off so it should be lowered as soon as the distillation is finished.


thermometer
thermometer adapter
still head
clamp
still pot
heating mantle
jack
keck clip
condenser
water out
clamp
water in
keck clip
vacuum adapter
keck clip
receiving flask
(supported in some way)


thermometer adapter
three way adapter
(still head)
3-prong clamp
round bottom flask
(still pot)
heating mantle
jack
clip
condenser
water out
clamp
clip
vacuum adapter
water in
water
source
collection
flask
sink
Text
24.4 °C

Procedure

Follow the steps exactly in the order below to set up a simple distillation of coffee.

- Expand a lab jack part way. Place a heating mantle on the lab jack and the lab jack on a ring stand base.
- Secure a clamp somewhere above the mantle. Tighten all screws except the prongs.
- Tighten the prongs around the joint of a **100 mL** round bottom flask. Lower the clamp (or raise the jack) so that the flask makes contact with the mantle. The still pot should be tightly secured now - lowering the jack should not move the flask.
- Add 35 mL of freshly brewed flavored coffee and one boiling stone to the still pot.
- Add a small amount of grease to the bottom joint of the still head. Rotate the joint in the still pot until there is an unbroken clear band at the top of the joint. *NO KECK CLIP here!!*
- Connect the condenser to the still head with a keck clip and support it with a clamp on a second ring stand. The clamp should be snug but not tight.
- Attach the vacuum adapter to the condenser with a Keck clip.
- Place a vial under the vacuum adapter. Support with a clamp or another jack.
- Insert a Vernier high temperature thermometer in the thermometer adapter and adjust its position (not while it is on the apparatus!).
- Connect the lower hose to the water (CW) nozzle, put the upper hose in the sink and start the water flowing.
- *Have your instructor check your setup.*
- Turn on the heating mantle to medium high. Stop the distillation when about 5 mL of liquid remains in the still pot. Do not let the still pot run dry.

Characterization

- Record the boiling range of the distillate (temperature a first drop collected, temperature when collection is stopped)
- Record the refractive index of your distillate and deionized water.
- Record the smell of brewed coffee, your final distillate, and the residue in the still pot.

Waste: All waste may go down the sink.

The Refractive Index

One of the most convenient methods of assessing a liquid's purity is to measure its refractive index. The refractive index is defined as the ratio of the speed of light in a vacuum to the speed of light in the medium in question:

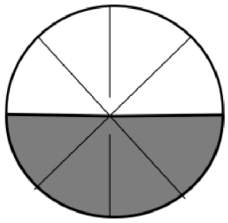
$$c_{vacuum}/v_{medium} = n \quad \text{(the refractive index)}$$

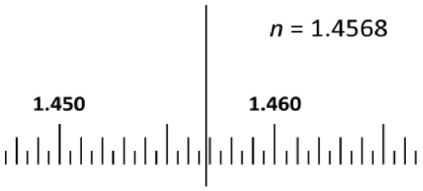
Since the velocities cancel and nothing travels faster than the speed of light in a vacuum, *n* is always a unitless number greater than 1. The refractive index of organic liquids is very sensitive to impurities. Impurities can make the refractive index higher or lower than the literature value. The refractive index also changes with temperature so it is important to compare samples taken at the same temperature.

Using the Abbe Refractometer

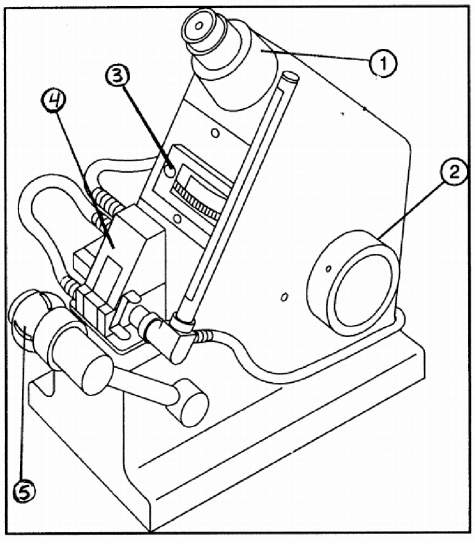
Never let hard objects touch the prisms or they will scratch!!

1. Open the prism and remove the kimwipe used to prevent scratching during storage.
2. Using a plastic pipette put several drops of the sample liquid on the bottom prism. Close the hinged prism and pivot the lamp up so that it shines on the sample.
3. Flip the toggle switch to the middle position to turn on the lamp.
4. Look into the eyepiece and turn the adjustment knob until you see a half moon image (dark semicircle on the bottom, light semicircle on the top). Line up the light/dark border with the intersection of the cross hairs.
5. If the light/dark borderline is not sharp, move the compensator dial until it is sharp. Use the adjustment knob to reposition the borderline exactly on the crosshairs.





6. Hold the toggle switch down and read the refractive index from the top scale to four decimal places. The small hashes are 0.0005, the medium hashes are 0.001. Release the toggle switch.
7. Open the hinged prism and clean with acetone and a kimwipe for the next use.
8. Close the prisms , placing a clean kimwipe between them and make sure the lamp is off.



1 – Eyepiece
2 – Adjustment knob
3 – Compensator dial
4 – Hinged prism
5 – Lamp