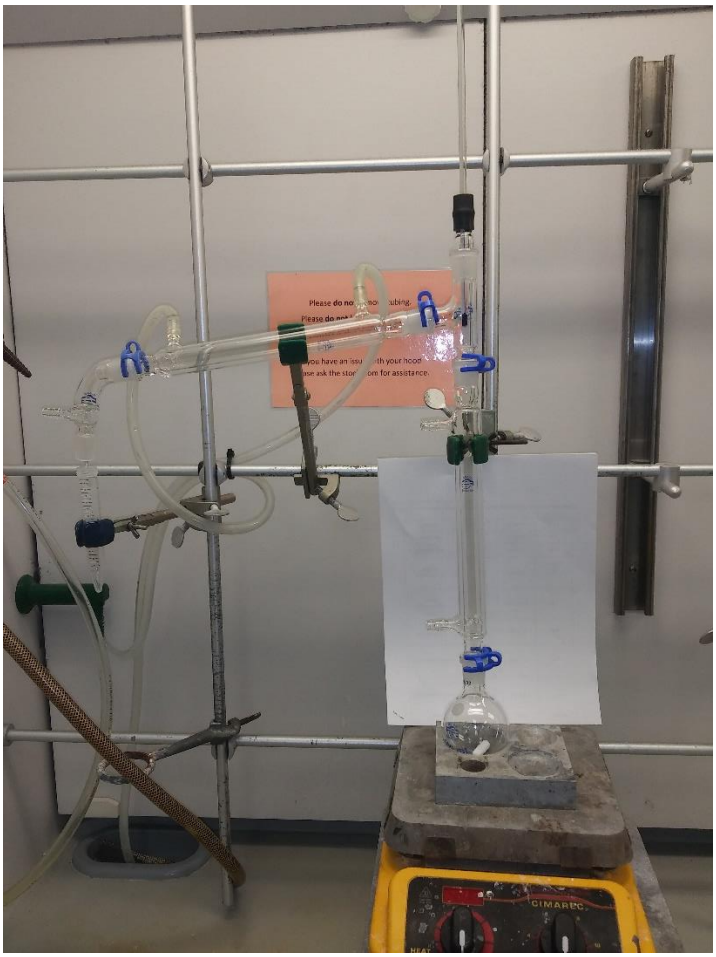
Fractional Distillation

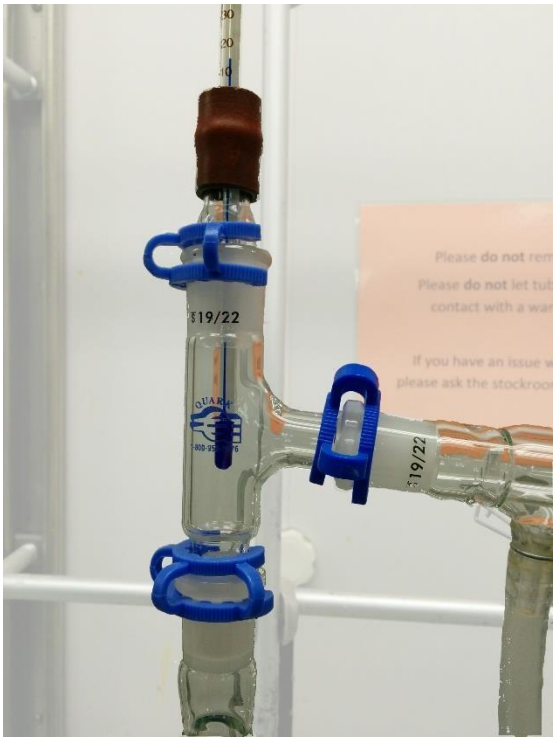
Apparatus Setup

The equipment setup for this lab will require the use of the following glassware assembled as shown:

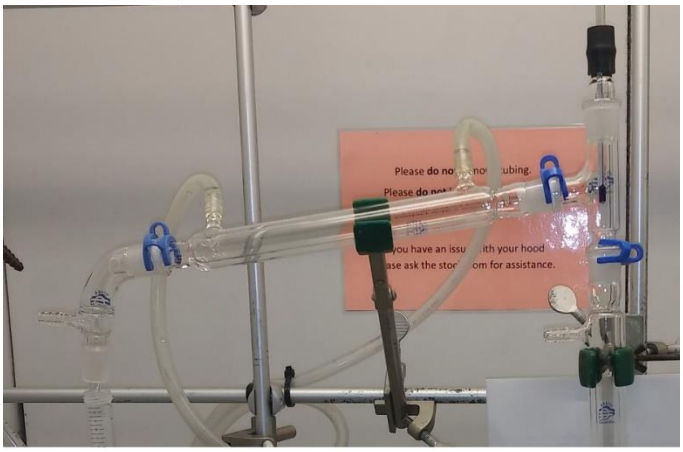


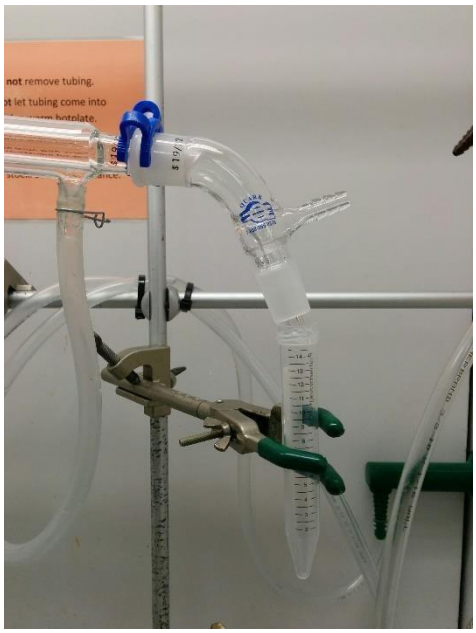
1. Place the vertical column over the correct indentation of the aluminum heating block (**the largest indentation**) and secure it to the scaffolding with a clamp. Be sure that the largest indentation is centered on the hotplate.
2. Connect the 3-way adapter and add a Keck clamp.
3. Get the straight adapter and put the rubber adapter over the rounded end. Gently slide the thermometer into the rubber adapter.
4. Place the straight adapter in the top of the 3way adapter and add a Keck clamp.

1. Adjust the thermometer such that the bottom of the thermometer is is line with the bottom of the arm of the the 3-way adapter.



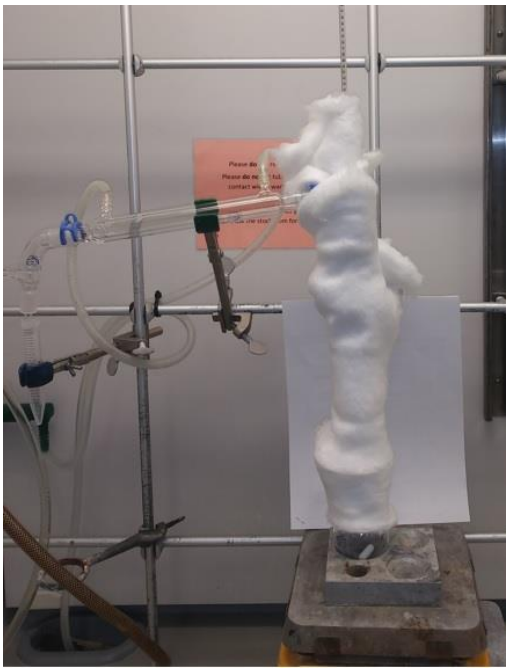
The horizontal portion of the apparatus should be assembled as such:

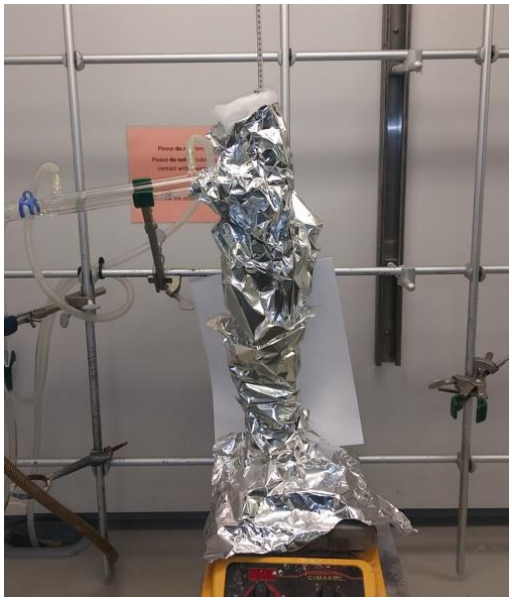




1. Acquire 30 mL of the unknown mixture in a graduated cylinder and pour into a 100 mL round-bottom flask.
2. Put a very, very small amount of grease on the bottom of the vertical condenser and slide on the round-bottom flask. Add a Keck clamp.
3. Raise the hot plate by turning the knob on the riser until the round-bottom flask rests snuggly in the aluminum block.
4. Loosely wrap the setup with cotton.
5. The loosely wrap the whole apparatus with aluminum foil.

1. Get the other condenser out of the case and attach the water tubes to the side arms. While the condenser is vertical with the rounded-end on top, be sure the water flows into the bottom and out of the top side arm.
2. Attach this condenser to the arm of the 3-way adapter and add a Keck clamp.
3. Clamp this second condenser securely using a metal clamp.
4. Connect the vacuum adapter to the end of the condenser and add a Keck clamp.
5. Position and clamp the first 15 mL centrifuge tube (**labeled fraction A**) below the vacuum adapter such that liquid flowing out of the adapter will flow into the falcon tube.





Procedure - Collecting Fraction A

Your unknown will contain 2 for the solvents listed below. You need to determine the identity of the two solvents and their ratio.

Table 1 Possible Solvents

Solvent	Boiling Point (°C)
Acetone	56.5
Hexane	68.8
Cyclohexane	80.7
Heptane	98.4
Toluene	110.6
Ethyl Benzene	136.2

Prior to performing the distillation, prepare a graph in your notebook for plotting the head temperature *vs.* the *cumulative* volume of distillate obtained. During the distillation, look for plateaus, collect three fractions, A, B and C, and record their respective volumes. Measure the amount of residual liquid in the distillation flask (if any) so that a % composition of distilled liquid can be calculated. Also record the boiling points of A and C.

Begin by heating the round-bottom flask. Turn on the magnetic stirring and turn the hotplate temperature to the temperature as directed from your TA (usually about 200 ºC). While the solution heats, insulate the vertical column and 3-way adapter with cotton and aluminum foil and insulate the round-bottom flask with just aluminum foil. Once the first drop of distillate drops into the centrifuge tube, start recording the temperature. As each additional mL of solution comes over into the centrifuge tube, check the thermometer and record the temperature. If the rate of the droplets adding into the centrifuge tube exceeds 2 drop every second, turn the hotplate down to 125 °C.
Eventually, the temperature will level off. This is the boiling point of the lower boiling compound in the mixture. There are 2 conditions that, if either is met, necessitate swapping fraction A for a new centrifuge tube:

1. The liquid is close to overflowing the centrifuge tube (or exceeds the graduated portion of the tube)
2. The temperature deviates from the plateau by more than 3 degrees C (higher or lower)

Once one of the conditions is met, swap fraction A for a new centrifuge tube, cap fraction A, and place the centrifuge tube in a beaker so that it stays upright.

Procedure - Collecting Fractions B & C

Once fraction A has been replaced with an empty centrifuge tube, turn the hotplate temperature up to 250 ºC. Continue collecting and recording the temperature as each mL of liquid comes over. Eventually, the temperature will plateau again. Once the plateau has been identified (2-3 mL of liquid come over at the same temperature), swap out fraction B for a new centrifuge tube, cap fraction B, and place it upright in the beaker with fraction A.

Continue collecting liquid until the rate of drops is very slow. Check the round-bottom flask periodically to ensure that there is still liquid in the flask. Do not distill to dryness, as peroxides may form, concentrate, and detonate. Once most of the liquid has been distilled (there will probably be about 3 mL remaining in the rbf, but that is ok). Cap fraction C and place with the other fractions. Turn off and lower the hotplate to allow the roundbottom flask to cool. Turn off the water in the condenser. Tell your TA that you have finished collecting the fractions and are ready to perform the GC analysis. Obtain GC plots for each of your fractions and carefully disassemble the distillation apparatus.