*WSU-Spring 2021 Organic Chemistry Survey Laboratory Prof. Thomas Nalli*

*Chemistry 340*

**Experiment #1 - Fractional vs Simple Distillation**

***Purpose*** *–* The purpose of this lab is to carry out both a simple and a fractional distillation of a 1:1 mixture of ethyl acetate and butyl acetate. The boiling point of the distillate will be closely monitored throughout each distillation and the distillates will be analyzed by gas chromatography – mass spectrometry (GC-MS) to determine the mole fractions of the two esters. This will allow comparisons to be drawn as to separation efficiency of the two methods.

***Simple Distillation****.* Start assembling the apparatus (*See Figure 13.7 p 149.)* by first clamping up a 25-mL round bottom flask to use as the distilling flask. Important - the top of the thermometer bulb should be even with the bottom of the side arm of the distillation head as shown in figure 13.7. A screw top vial will be used as the receiver and a sand bath as the heat source. Use a lab jack under the sand bath so that heating can be quickly discontinued if necessary.

When ready to begin the distillation, remove the thermometer and add 5 mL of each ester as well as a couple boiling chips. Begin heating with the sand bath voltage regulator set at 50%.

After boiling begins watch carefully for a reflux line, which then should rise up the neck of the flask and shortly after distillate should begin dripping into your receiving vial. Decrease the heat if necessary (raising the flask a bit out of the sand is the quickest way to accomplish this) to keep the rate of distillation from being too fast for you to be able to count drops and read the temperature every 5th drop. (20 drops ≈ 1 mL).

Divide the distillate into three portions by changing the receiving vial to a new one when the temperature reaches 85° and changing it again at 115°. The changes should be made quickly and without removing the round bottom flask from the sand bath. After making each change, cap the just removed vial and label it with your name, lab section, and identity (use a simple code such as S-1 for first simple distillate, S-2 for 2nd simple etc.). Later, weigh the labeled vials (without corks for future simplicity) and save for GC analysis.

Continue the distillation until about 1-2 mL of liquid remains in the distilling flask.

***Fractional Distillation.*** The apparatus for fractional distillation (*See Figure 13.17 on p 160)* is otherwise the same as that for simple distillation but adds in a fractionating column in between the rbf and the distilling head. Again, the first step in constructing the apparatus should be to clamp up the rbf above a lab jack. Add 5 mL of each of the compounds to be distilled and a boiling chip, then attach the column and continue to build the rest of the apparatus using Keck clips to hold the pieces together and a clamp on the condenser to keep the unit from tipping over as shown in the diagram.

Begin heating and watch for boiling and then for a reflux line. Try to increase the applied temperature in such a way that the reflux line moves slowly up the column. Once distillate begins collecting do not vary the heat input - only do so if necessary to keep the distillation rate at less than 2 drops per minute. Once distillation is proceeding, a piece of aluminum foil wrapped around the column is useful to prevent heat loss from the column.

As before record the temperature as a function of number of drops of distillate and change receiving vials at 85° and 115°. Make sure to label the vials, weigh and save for GC as above.

In the fractional distillation, you may notice a temperature drop about halfway through and a coinciding slow down (or stopping) of the distillation (distillate stops dripping into the receiving vial). This is a good sign! Add more heat to get the higher boiling butyl acetate up the column and proceed as before, stopping when about 1-2 mL of liquid remains undistilled.

***Analysis of Results.*** Prepare GC-MS sample solutions of your fractions S-1 and F-1 by adding 1-2 drops of the liquid to a new vial containing 5 mL dichloromethane. Transfer enough of this solution to half fill one of the tiny GC-MS sample vials that will be provided. Use a Sharpie to label the vial with your initials and the fraction number. (A few groups will also be asked to prepare GC-MS samples of the other fractions and that data will be shared with the rest of the class.)

Dispose of the distillates in the waste solvents bottle in the hood. Rinse the vials with acetone and allow them to dry (do not remove the labels). After drying, weigh each of the empty vials and determine the mass of each fraction of distillate.