# Organic Chemistry 211 Laboratory <br> Experiment 5b: Fractional Distillation 



In this experiment fractional distillation will be performed in order to separate ethyl acetate from butyl acetate. Also, a comparison should be discussed with the simple distillation experiment performed previously. Assemble the apparatus as shown in the diagram. Note that its only difference from the simple distillation is the addition of the Distillation Column. To construct the column, take the air condenser and fill it up with glass beads.

Add 20 mL of the mixture which contains unknown amounts of ethyl acetate and butyl acetate (the same mixture that you used for simple distillation) and one boiling stone to the distillation flask. Begin the heating with the heating mantle. Since butyl acetate is relatively a high-boiling (low vapor pressure) substance, it may be appropriate to initiate the heating on the maximum voltage
setting. Watch the distillation flask to see that it does not boil so vigorously that the liquid runs over the top. On the other hand, if due to inefficiency of heating, the vapors do not reach the take-off point in order to be distilled, a wrapping of alumina foil around the column and the distillation flask is advised. This is to transport additional heat from the heat source onto the higher points of the apparatus. Distillation should be dropwise. Record the temperature on the thermometer for every 0.5 mL of the distillate received. Later, make a graph (in your laboratory notebook) of temperature versus the volume of the distillate. Continue distillation for two or three more data points on your graph after a temperature plateau is reached close to the boiling point of butyl acetate (for graphing purpose). Important: Do not allow the round-bottom distilling flask to go dry.

For later GC analysis: During the distillation, you will collect 3 fractions according to the chart below and store each of them in a capped vial for the next experiment (Experiment 6: GC). You need not collect an exact volume. Record the exact temperature range and volume for each fraction you collected. Parafilm tightly each vial for storage.

|  | $1^{\text {st }}$ <br> fraction | $2^{\text {th }}$ <br> fraction | $3^{\text {th }}$ <br> fraction |
| :--- | :---: | :---: | :---: |
| Temperature range <br> $\left({ }^{\circ} \mathrm{C}\right)$ | $76-80$ | $88-92$ | $>110$ |
| Volume (drops or mL) | $\sim 1 \mathrm{~mL}$ | $\sim 1 \mathrm{~mL}$ | $\sim 1 \mathrm{~mL}$ |

## Disposal Note:

- At the end of the experiment, leave the glass beads in the half beakers inside the disposal hood and let them dry up. DO NOT throw away the glass beads!
- Boiling stones should not be dumped into the sink with the liquid. Throw them away into the regular trash.


## For the report:

$>$ You should write one combined lab report for both Simple Distillation and Fractional Distillation experiments.
$>$ Two graphs are required: (do graphs horizontally on paper)
(1) One graph of Temp (y) vs. Volume (x) (for both simple and fractional distillations on the same graph; i.e. two sets of data plotted on the same graph);
(2) One graph of Time (y) vs. Volume (x) (for both simple and fractional distillations on the same graph). Remember to label axes.
$>$ Explain how and why the graphs for the two types of distillation are different.
$>$ Discuss the theory in details using the words including vapor pressure, boiling point, reflux, azeotrope, and theoretical plate. Compare and contrast the two types of distillation.
$>$ Why would temperature drop in the middle of the fractional distillation while heating is continuously provided?

