

Exp. 9, Separation by Simple and Fractional Distillation And Analysis by Gas Chromatography

LEARNING OUTCOMES:

After performing this experiment the student will be able to:

1. Perform a simple distillation and a fractional distillation
2. Obtain a gas chromatogram on a sample and interpret it
3. Explain the difference between the separation of a simple distillation and of a fractional distillation and the reasoning behind it
4. Describe how separation is achieved in gas chromatography

INTRODUCTION:

This experiment is a technique lab on separating and purifying compounds by distillation and on using GC to analyze samples. Distillation is a frequently-used technique involving boiling and condensing and is used for mixtures of liquid compounds that have different vapor pressures (different boiling points). When a liquid mixture is boiled, the vapors above the liquid are richer in the lower-boiling component. Condensing these vapors results in a purified sample. Figure 1 shows a typical laboratory setup for a simple distillation.

When the difference in boiling points of the components is large ($>40\text{-}60\text{ }^{\circ}\text{C}$), a fairly good separation often can be made with a simple distillation (Figure 2b). When the difference in boiling points of the components is small, then a simple distillation cannot achieve a good separation (Figure 2a). If a better separation is desired when the components have similar boiling points, then a fractional distillation can be done. The simple distillation setup in Figure 1 can be converted to a fractional distillation setup by inserting a fractionating column at number 11 in the figure. The fractionating column can be thought of as a place where the vapors condense and then boil again. Each time a very small sample condenses and then boils again, the vapor is even more enriched in the more volatile (lower boiling) component. Thus, fractional distillation can produce samples that are much purer. This increase in purity comes with a cost of more complex equipment and more distillation time.

Gas chromatography (GC) is another separation technique. Since the amount of sample that can be separated at one time is very small, it rarely is used for purification of material. It is used quite often in analyzing mixtures. In GC a sample in the gas phase is passed through a column (stationary phase) with a flow of a gas (mobile phase). Different compounds will be retarded different amounts by different interactions with the stationary phase, a coating on the inside of the column. With the column we are using many compounds pass through the column in order of increasing boiling points, and is sometimes called a "boiling point column". However, the compounds are not separated by boiling points but are separated by a combination of the vapor pressures of the components and how much each interacts with the stationary phase.

This experiment will be done in teams of two, with each team setting up and tending a simple distillation and a fractional distillation. At specific volumes the temperature will be measured and a sample collected. Each team

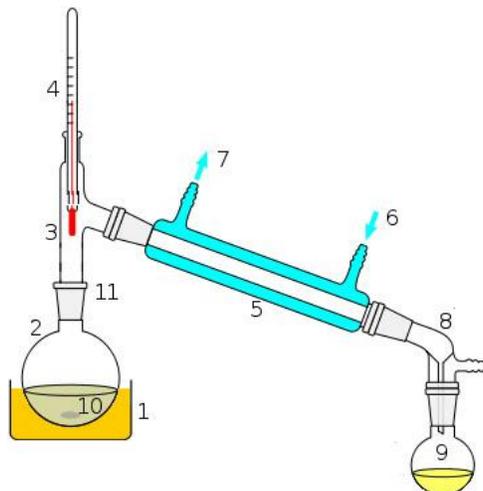


Figure 1. Laboratory display of distillation: **1:** Heating mantle **2:** Still pot **3:** Still head **4:** Thermometer **5:** Condenser **6:** Cooling water in **7:** Cooling water out **8:** Vacuum adaptor **9:** Receiving flask **10:** Anti-bump granules **11:** Expansion place for inserting a fractionating column.

Adapted from http://en.wikipedia.org/wiki/File:Simple_distillation_apparatus.svg

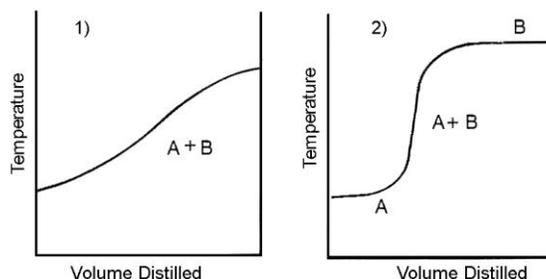


Figure 2. Distillation curves for a) poor separation and b) good separation.

will have its own gas chromatograph (GC), on which the samples will be analyzed. The results of the two different types of distillation will be compared.

PRE-LAB:

Be sure you include everything in your pre-lab as described in the handout on the first day.

HAZARDS:

Pentane, ethyl acetate and propyl acetate are quite flammable, are harmful if swallowed, and can cause eye, skin, and respiratory tract irritation.

EXPERIMENTAL PROCEDURE:

The two students sharing a hood will act as a team. One student will assemble a simple distillation apparatus and the other student will assemble a fractional distillation apparatus. Assemble the apparatus shown on the instructor's desk, using a 50-mL round bottom flask as the distilling flask and a 50 mL graduated cylinder as the receiving container. Observe the placement of the two plastic joint clips. Carefully note the position of the thermometer in the figure. The sensing area of the thermometer must be placed below the sidearm, or it will not read the temperature correctly.

Place several boiling stones into 50-mL round bottom flask. Then add 25.0-mL of the mixture of 50 mole% ethyl acetate and 50 mole% propyl acetate from the repipettor to the flask and finish assembling the apparatus.

Turn up the heat so that the liquid starts boiling soon. While it is heating turn on the condenser water to a slow flow. As the solution starts boiling turn down the heat so that the liquid is boiling moderately vigorously. After 10-20 minutes of heating the temperature should rapidly rise and liquid appear in the condenser.

During this time prepare the sample vials for GC. If your team is using a mini GC, get 13 screw-cap vials, put 1.0 mL of pentane in each, and put caps on them. Use a marking pen and label six of the vials S 0.1 mL, S 5 mL, S 10 mL, S 15 mL, S 20 mL, and S last. Label another six similarly, but with an F for fractional distillation instead of an S for simple distillation. If you are using a regular GC, get 15 screw-cap vials, put 1.0 mL of pentane in each, and put caps on them. Use a marking pen and label the vials F 0.1 mL, F 4 mL, F 8 mL, F 12 mL, F 16 mL, F 20 mL, and F last. Label another seven similarly, but with an F for fractional distillation instead of an S for simple distillation. (Most modern gas chromatographs are fairly sensitive, requiring the distillation sample to be diluted. We will be diluting with pentane.) Label the remaining vial 50/50 and put 50 μ L of the 50 mol% ethyl acetate/50 mole% propyl acetate, and shake.

For the rest of the experiment one member of a team will tend both distillation setups, while the other member will do GC. About half way through the distillations the team members will switch roles. The GC member can record the gas chromatogram of the 50/50 sample before any distillates have been collected and will record the GCs' of the samples as they come from the distillations.

It is important to regulate the rate of heating so that the distillation occurs at a rate of about 1 drop per 3-4 seconds. At the proper rate it should take you 40-60 minutes to do the entire distillation. If the distillation is performed more rapidly than this, you will not achieve as good of separation between the liquids. About half way through the distillation with the fractional distillation column have the instructor help you put aluminum foil on your distilling flask to decrease heat loss.

When the first drop appears at the end of the condenser, record the distillation temperature as a volume of 0.1 mL. Collect the next 3-4 drops of distillate in the sample vial labeled 0.1 mL. Collecting 3-4 drops can be difficult. Do not touch the vial to the glass of the condenser. Be sure the drop goes into the vial and not down the outside. Put the cap on and shake several times. Resume collecting the distillate in the graduated cylinder and recording the temperature at every 1.0 mL of distillate collected in the 50 mL graduated cylinder. Record your data in the table below.

As the distillation progresses the temperature will increase, turn up the heating mantle to maintain the same rate of distillation. When you have collected a total volume of distillate that matches the volume on your next sample vial, take another sample as above. Continue taking samples until the last sample. This sample will be collected when the volume in the distilling flask is about 2 mL. Be sure to record the temperature and the total volume of distillate at this sampling. Then stop the distillation by turning off the heat and immediately lowering the heating mantle. Do not distill to dryness – the flask may crack!

When you are done with the experiment empty your six GC samples into the small container labeled GC wastes. Then put the material you distilled into the container labeled ethyl acetate/propyl acetate distillation mixture to be reused. DO NOT put any of your pentane samples in this container.

LAB REPORT:

The lab report for this experiment will be the completed pages below. Open this document in a word processor and answer the questions and fill in the blanks. Expand the spaces between the questions as necessary. Do not hand the pages before the report as part of your report.

Below are several comments to help you understand distillation better.

A) In a distillation the composition of the vapor phase is different from the composition of the liquid phase (what is in the in the distillation flask). The vapor phase has more of the substance with the lower boiling point (greater vapor pressure) in it. This is what allows for separation by distillation.

B) The greater the difference in boiling points, the higher the percentage of the more volatile substance will be in the vapor phase.

C) A fractional distillation is, in effect, multiple distillations in the same setup. The first distillation is in the distillation flask. The other distillations are in the fractionating column.

D) The GC detector responds differently to each compound. Thus, the areas of the peaks are not directly proportional to the numbers of moles when you're comparing two different compounds. (Do remember, however, that in NMR each hydrogen nucleus contributes the same amount to the area of a signal regardless of whether it is in the same compound or in different compounds.) Response factors (relative response factors) are used to compensate for the different detector responses. In the equation below the _____ is the response factor. The equation below is used to calculate mole% of propyl acetate from your experimental data.

Exp. 9 Report for Separation by Simple and Fractional Distillation

Name _____ Section _____ Date _____

1. Add the temperature data from both distillations to the table to the right. Then construct, using a spreadsheet or a graphing program, a graph of temperature vs. mL of distillate with two curves on it. One curve will be your simple distillation data while the other will be the given fractional distillation data. Be sure you use proper graphing techniques, e.g. title, labels and units on axes, legend, reasonable size and scale, etc. Draw in a curve that you think best represents your data and a curve that best represents the fractional distillation data. This curve should not be a dot-to-dot line. This curve should be drawn by hand and NOT by computer. Use the distillation curves in Figure 2 to help you estimate the curves.

Volume Distillate, mL*	Simple Distillation		Fractional Distillation	
	Temperature, °C	Mole % Propyl acetate**	Temperature, °C	Mole % Propyl acetate**
0.1				
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
*Assume that 0.1 represents the first two or three drops				
**Mole % of Propyl Acetate in Distillate Sampled at this Volume, determined using GC data.				

2. Use the graph prepared in question 1 and explain what these curves say about the effectiveness of the simple distillation compared to the fractional distillation and explain how it says it.

3. Calculate the mole percent propyl acetate from both of your distillations and add these values to the table above. See the comments above for how to do this calculation. Then construct, as in question 1 above, a graph of mole% propyl acetate vs. mL of distillate with two curves on it. One curve will be your simple distillation data while the other will be the given fractional distillation data. Again, use proper graphing techniques.

Draw in a curve that you think best represents your data and a curve that best represents the fractional distillation data. This curve should not be a dot-to-dot line. This curve should be drawn by hand and NOT by computer. Use the distillation curves in Figure 2 to help you estimate the curves.

4. Does this graph show the same information as the temperature graph of question 2? Does it show it any better or poorer?

5. Paste your two graphs into this report near this position, rearranging somewhat if necessary to avoid a lot of white space. Size each graph so that it is the full width of the page and less than $\frac{1}{4}$ of a page in height. You do not have to associate captions with the graphs.
6. Fill in the following table and then answer the question below.

Literature boiling point of ethyl acetate, °C	Literature boiling point of propyl acetate, °C	Reference for literature values (Do not use an MSDS sheet as a boiling point reference.)

- a) Are either of these two boiling points on your simple distillation graph or the fractional distillation graph? If so, explain where. If they are not, explain why they are not on the graphs.
7. Assume you are starting with the same ethyl acetate/propyl acetate mixture used in lab, and with the same starting volume. Also assume you are doing the fractional distillation as described in this experiment. How could this apparatus for the fractional distillation be modified to get a larger volume, and at a greater purity, of each the two different compounds?
8. Explain how intermolecular forces are important in gas chromatography and how they affect retention times.