

11. Fractional Distillation

Distillation, another method of separation, is the most commonly used method of purification for liquid samples. You will perform fractional distillation in this experiment. As part of the distillation process, you will obtain the boiling point range of both components of your mixture. The better your separation, the more accurate your boiling points will be. You will obtain indices of refraction of your samples after the distillation.

Both boiling point and refractive index are physical properties that are used to characterize organic liquids and give some indication of their purity. You will use the boiling points and indices of refraction to assess how well the mixture was separated using each method of distillation.

PRE-EXPERIMENT ASSIGNMENT

Study this chapter of the manual, the lecture notes on the Chemistry Department web site, and pages 72-74, 76-78, 80-91, 98-100, and 110 in Williamson. Do the first parts of your notebook writeup.

A student who has prepared for the Fractional Distillation experiment should be able to:

1. Perform all of the tasks listed under the Boiling Point and Index of Refraction experiment.
2. Define, and explain the difference between, simple and fractional distillation.
3. Define, recognize, and explain: reflux, theoretical plates, azeotrope.
4. Give a drawing of, and identify the components from a drawing of, the apparatus used for simple and fractional distillation. Give the function of the components, and predict the consequences of improper assembly and use (including thermometer bulb placement, distilling to dryness, and closed systems).
5. Draw the structure given the name, or give the name from the structure, of the compounds used in the day's experiment, and give the role of each (reactant, solvent, catalyst, etc.).
6. Identify and explain safety considerations for this experiment.
7. Perform the day's experiment safely and successfully.

Quizzes given after the experiment has been performed may also include:

8. Predict results of experiments using simple, fractional, and vacuum distillation, and decide which of the three is best for a given sample from the necessary data about the sample.
9. Draw and interpret distillation curves; for example, determine boiling

points and distinguish between simple and fractional distillation from these curves.

Fractional Distillation

Turn your sand bath on and set it to about 30 or 40. Later you will probably have to adjust this setting. While waiting for the sand bath to heat up, assemble the apparatus shown in fig. 5.6 of Williamson. The apparatus should be set up above the sand bath and lowered into place when you are ready to start heating. The flask is the 5 mL short-necked flask from your kit. Place a column containing a copper sponge between this flask (the 'pot') and the distillation adapter. Put a boiling chip and a mixture of 2 mL of toluene and 2 mL of cyclohexane in the flask.

Be careful when setting up and taking apart the apparatus. Be firm but gentle, especially with the thermometer. The placement of the thermometer is critically important. A good rule of thumb is that the top of the mercury bulb should be even with the bottom of the side arm of the distillation apparatus. It should not be too high or too low. The end of the distilling head should fit well into the receiver. The receiver should sit on the bottom of a 50 mL beaker filled with ice (not shown in the figure). Have a second receiver ready to replace the first one. Be careful not to allow the ice to get into the receivers.

When everything is assembled and ready to go, scoop an indentation in the hot sand using your spatula or scoopula. Lower the apparatus so that the bottom of the distilling pot (the 5 mL flask) is just touching the surface of the sand in the indentation. You can control the heating rate by piling sand around the pot or scraping it away with your spatula. Don't expect the temperature on the thermometer to begin rising immediately. It will not start to register an increase until vapors from the boiling liquid reach the mercury bulb. This will take several minutes.

The distillation process should be relatively slow. Adjust the rate of heating by manipulating the sand in the sand bath as described above so that the rate that drops enter the receiver is no faster than two drops per minute. The slower the distillation is the better the separation will be. Record the thermometer reading each time a drop enters the receiver. (This means that you should end up with a table that tells you that drop 1 appeared at a certain temperature, drop 2 appeared at a certain temperature, and so on. It does NOT mean that you should end up with a table that tells you that so many drops appeared at one temperature, so many drops appeared at another temperature, and so on.)

After you collect about one milliliter in the first receiver, switch to the second receiver. Do not stop the distillation to do this and keep recording the temperatures as before. All of these data go into the same table--do not start a new table when you switch receivers. Continue distilling until only about 0.4 mL of liquid remains in the flask. To help you estimate that amount, remember that it's about one tenth of the starting volume.

If you stop too early, you will not have enough data for a good distillation graph. The temperature at the end of your experiment should be at least 105°C and preferably more, unless your pot is about to distil to dryness before this happens. ***You must stop while there is still liquid in the pot.*** Distilling to complete dryness is dangerous because many organic liquids contain trace amounts of peroxides that can explode when heated (see Williamson, chapter 2).

Measure the index of refraction for the first fraction (receiver 1) and for the residue remaining in the pot.

CLEANUP

Excess distillation mixtures and products go in the non-halogenated liquid waste container in the hood. Used Pasteur pipettes go in the broken glass box.

POST-EXPERIMENT ASSIGNMENT

Write the lab report and have it ready to turn in by the beginning of the next lab.

Your report must include a distillation curve similar to that in figure 5.9 on page 91 in Williamson, prepared from the data you collected. In place of mL for the x axis, use drop number. (Remember that you are plotting the temperature at which each drop appeared, ***not*** how many drops appeared at each temperature.) Draw the best fitting smooth curve that fits the data--do not "connect the dots."

Use the figure as a guide to the type of curve that you should expect. Don't expect all of your points to fall exactly on the curve, however. The figure is unrealistic in that respect. The boiling point of each component is obtained from observing the relatively flat areas on the curve.

You may either plot graphs by hand or use a spreadsheet program on your computer. If you plot your curves by hand, use good quality graph paper, and use the procedure you learned in General Chemistry. See the Chemistry Department web site for instructions on how to use a spreadsheet program.

In the Conclusions section of your report, discuss the success of your distillation. Does your curve show that your compounds were effectively separated? Compare your curve to the curve for a good separation shown in Williamson. Also, compare the refractive indexes that you obtained to those of the pure compounds. Are the liquids in the first receiver and the pot pure or nearly so? Prepare for the distillation portion of the next quiz.