

Fractional Distillation

Organic Lab – AEMoody

Source: AEM Handout, updated 9/27/07

The distillation technique exploits differences in boiling points to separate materials. In a simple distillation, a liquid is vaporized, condensed, and collected. The collected material is then called the distillate. For practical purposes, the two components of a simple distillation should have boiling points that differ by more than 100°C to give effective separation. In a fractional distillation, many simple distillations are performed in series on a fractionating column, allowing materials with much more similar boiling points to be separated from each other.

Note that we will be working with a rather unusual material in this experiment: an azeotrope. An azeotrope is a mixture that distills with a constant boiling point and at a constant composition. In this case, ethanol and water form an azeotrope with a composition of 95.57% ethanol by weight, but commonly called “95% ethanol”.

Experimental:

You should turn in a 125 mL Erlenmeyer flask to be filled with about 100 mL of ethanol and water, combined in unknown proportions. Your task during this lab is to fractionally distill this mixture; by observing the temperature and volume of the distillate, you can determine the proportion of ethanol in the mixture.

When you arrive in lab, obtain a macroscale glassware kit and assemble the fractional distillation apparatus as shown in your text. Measure the volume of the EtOH/H₂O mixture that you will distill using your graduated cylinder. Record the total volume that you use. Pour the mixture into a 250 mL distillation flask with a couple of boiling chips. (You should always use a round-bottomed flask that holds about twice the volume of the total solution that you are distilling.) Grease lightly the connections between the ground glass joints. We will also use metal sponge packing in the fractionating column. Be sure to construct your apparatus so that the fractionating column is vertical from all perspectives, and that no gaps are evident at any of the joints. Hold the entire construction together using clamps and blue clips. You should support the heating mantle containing the round-bottomed distillation flask with your lab jack so you can remove the heat as needed. Substitute a 10 mL graduated cylinder for the round-bottom flask as the receiving flask, so that you can monitor the volume of the distillate as you collect it. To most accurately monitor the temperature, the thermometer bulb should be positioned just below where the distillation head bends toward the condenser. The lab instructor or TA should check the assembly before you proceed.

Begin heating the mixture slowly. Soon, you should see a ring of condensed vapor rising up the fractionating column. Slow steady rise of this ring and slow steady collection of the distillate will result in the best separation. Once you are collecting distillate in your graduated cylinder, a rate of one mL of distillate per minute is often about right. If it takes 20 drops to make one mL, that that is a rate of 1 drop every three seconds. However, you may choose to distill more slowly so as to obtain better separation of the ethanol from the water. Again, try to keep the distillation rate slow and steady, while still finishing the lab on time.

Record the volume of the distillate and the distillation temperature throughout the process. The temperature readings can be taken every mL at the beginning and at the end of the distillation, but they should be taken more frequently (as fast as you can observe them) when the boiling temperature is changing rapidly. You may want to dump all of the fractions into a beaker until the whole process is over, but ultimately, these materials can all go down the drain.

After the distillation temperature has remained steady at the highest expected boiling point for 3-5 mL, stop the distillation. Never carry out a distillation to dryness! Graph your data (as an x-y plot) of temperature vs. volume of distillate. Add a smooth curve by hand to this graph, since it is not easily fit by a computer function. Estimate the inflection point of the curve on the graph and note the volume at the inflection point. This volume should be equal to the volume of 95% ethanol in your original sample. Using that volume and the original total volume of the EtOH/HOH mixture, determine what percentage of ethanol was in your original sample. Show your work in your notebook! (Note: you will probably not have the same percentage as your neighbor.)