Fractional Distillation

Organic Lab – AEMoody

The distillation technique exploits differences in boiling points to separate materials. In a simple distillation, a liquid is vaporized only once, 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; this mixture is commonly called "95% ethanol".

Experimental:

You should first 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 will determine the proportion of ethanol in your mixture. (Note: you will probably not have the same percentage as your neighbor.)

Next, obtain your *macroscale* glassware kit and assemble the fractional distillation apparatus shown to you in your recitation. You will need these pieces: a roundbottom flask, a fractionating column (the "fat" condenser that you will pack with copper sponge packing), a distillation head, a thermometer adapter, a thermometer, a water-cooled condenser, and an adapter. Grease lightly the connections between the ground glass joints. Be sure to construct your apparatus so that the fractionating column is as vertical as possible from all perspectives, and that no gaps are evident at any of the joints. Hold the entire construction together using clamps to the monkey bars and blue clips on the ground glass joints.

Measure the volume of the EtOH/H_2O mixture that you will distill using your 50 mL graduated cylinder. Record the total volume that you use. Pour the mixture into the roundbottom distillation flask with a couple of boiling chips. (You should always use a round-bottomed flask that holds at least twice the volume of the total solution that you are distilling.) You will heat the distilling mixture in the roundbottom flask using a sand bath in your larger heating mantle. Bury the flask deep in the sand so that you achieve good heat transfer, and support the sand bath with your lab jack so you can remove the heat as needed. Use your 10 mL graduated cylinder 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. Turn on the water to cool the water-cooled 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, then 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 (on the y-axis) vs. volume of distillate (on the x-axis). 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 is 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 calculation in your notebook!