Chem 267: Distillation (revised 6/2020)

Liquid substances are most often purified by distillation. In the first part of this experiment, to practice the technique, you will separate a mixture of cyclohexane and toluene (tol-u-ene) by fractional distillation. In the second part of this experiment, you will distill an unknown mixture and (1) determine the identity of the components by observing their boiling points (BPs), and (2) determine the relative amounts of each by measuring the amount of distillate collected at each BP. The purity of each unknown will be checked by gas chromatography (GC). The most difficult part of distillations is setting up the small apparatus properly and attaining a slow, steady heating rate. Take your time to do it correctly. You will use this technique in several experiments later on.

Do Prelab Exercise a.) on p. 86 as part of your prelab outline. Note that the procedure you will follow is the one given below on this handout and not that given in the Distillation chapter of the lab text. The lab text should be used as a reference.

Prelab: You may either print out your prelab and bring it with you to lab, or bring your computer. Your TA will grade it on the spot before you begin the experiment. For the in lab observations, you may use scratch paper and record later in your ELN, or bring your computer and record directly in your ELN.

Postlab Report: Make sure to use the non-formal postlab report template on the course website!

Fractional distillation of Cyclohexane and Toluene: The distilling column is packed with a copper mesh to aid in the separation and to increase the number of theoretical plates. Because the distilling path is longer, heat loss is greater in fractional distillation, so it is helpful to wrap slightly crinkled aluminum foil loosely around the column to insulate the column from drafts and to help keep the heat in. This helps to provide the heat necessary to distill the material through the apparatus at a steady rate. In the write-up, plot the temperature vs # of drops.

Fractional Distillation of an Unknown Mixture: It is not necessary to wash the apparatus or to change the copper sponge. These items are contaminated only with volatile (easily evaporated) compounds. Simply dry the apparatus in the fume hood by blowing air gently over and through the glassware to evaporate most of the remaining cyclohexane and toluene. The fractionating column may be dried more easily by drawing air though it by connecting it to the vacuum. You will be assigned to distill 4 mL of a mixture (measure it) of two of the compounds given in the following table.

compound	**boiling point (BP) (°C)**
acetone	56
methanol	65
hexane	69
2-methyl-2-propanol	82
heptane	98
toluene	111
1-butanol	117

In the distillation of the unknown mixture, after the first component has distilled over its temperature plateau (± a few degrees) and the temperature begins to rise more steeply, the collection vial should be changed to prevent contamination of the first component with the second. The material collected between the BPs of the two substances is a mixture of the two and should be considered to be an impure fraction. Once the temperature reaches a second plateau, this should be considered to be the BP of the second substance and the collection vial should again be changed to collect pure component number two. Thus, three vials of distillates will be collected: one containing pure lower-boiling component, one containing the intermediate mixture, and one containing pure higher-boiling component. Using the BPs and amounts of each pure fraction, determine the identities and ratios of the unknowns. As in all distillations, because a small amount of material is purposely left behind in the flask and because of holdup in the apparatus, the amount of the second component that is actually collected in the collection vial will be less than the amount in the original mixture. To

determine the ratio in the original mixture, use the total amount of original mixture (4 mL) and the amount of the first component collected to determine the amount of the second component. Using GC, check the purity of the lower-boiling fraction of the unknown mixture.
Collecting separate fractions is not done in the distillations of cyclohexane and toluene because these experiments are simply done to learn the operation of distillation. In distillations in general, however, once the first fraction has distilled, the vial must be changed to collect the intermediate, impure fraction; then, once the second pure component begins to distill, the vial must be changed again to collect that pure component separately.

SAVE THE COPPER WIRE AND LEAVE THE COPPER SPONGE IN THE COLUMN. THESE WILL BE USED IN FUTURE EXPERIMENTS.

WASTE: Place all liquids into the NONHALOGENATED liquid waste container.

Things to Watch Out for in Distillations:

(1) The thermometer is positioned incorrectly. This leads to observed temperatures which are incorrect. The top of the mercury bulb must be even with the bottom of the side arm on the distilling head. It must not touch the copper packing or the glass apparatus.

(2) Distillation is too rapid due to excessive heating. This leads to a poor separation. Start over if this occurs.

(3) Not enough heating. This leads to reflux (a condition in which the vapors condense and return to the distilling flask) instead of distillation. Supply enough heat so that the distillation proceeds steadily at a rate of about one drop per 20 - 30 seconds.

(4) After the low-boiling fraction has distilled, the distillation may slow or stop and the temperature may fluctuate or drop. This is because more heat is required to distill the higher-boiling fraction. Lower the apparatus, or adjust the temperature setting so that the distillation proceeds at a rate of one drop per 20 - 30 seconds. It is advisable, especially if the lab is drafty, to insulate the distilling column in the fractional distillation with loosely wrapped aluminum foil.

(5) The distilling column is packed too tightly with copper sponge. This leads to a situation known as flooding of the column, in which a plug of liquid collects in the column. Distillation will be severely hampered in such a case.

(6) The distilling column is packed too loosely. A poor separation results.

(7) Vapors are lost through poor connections. Be sure that the connectors are in good condition and that the apparatus stands vertically so that strain is not placed on any of the connections.

(8) With this equipment, a measured BP can be considered to be reliable only after the temperature has leveled off and the distillation rate is steady and about 1 drop per 20-30 seconds. Even so, the temperatures may be off by a few degrees.

Postlab Questions

1.) From the boiling point vs volume plot for your fractional distillation, what can you conclude about the purity of each of the two components in the distillate?

2.) Refer to Fig. 5.8 in the lab text. How much pure cyclohexane can be isolated in the fractional distillation shown? Show a sketch of how you determined this.

3.) Refer to Fig. 5.3 in Williamson. Starting with a mixture of 10% cyclohexane and 90% toluene, to produce distillate having a purity of greater than 90% cyclohexane, what is the minimum number of theoretical plates that a fractional distillation apparatus would need? Show a sketch of how you determined this.

4.) If the distillation of cyclohexane and toluene were carried out while a hurricane raged outside what effect would this have on the boiling points (hint: atmospheric pressure. See p. 56)?

5.) What is the single most important variable that contributes to an effective separation in a distillation?