EXPERIMENT #6
Distillation and Refractometry

Distillation is a common technique used to separate components of a liquid mixture based on differences in boiling points of the liquids. We will explore two different types of distillation (simple and fractional) and determine which type gives better separation of our liquid mixture. Then we will use refractometry (a measurement of how much light is bent as it passes through a liquid) to identify the components of our mixture.

READING ASSIGNMENT:

Technique 13: Boiling Point and Distillation in Techniques in Organic Chemistry 3rd Ed.
Technique 16.3: The Refractometer in Techniques in Organic Chemistry 3rd Ed.
Technique 16.4: Determining a Refractive Index in Techniques in Organic Chemistry 3rd Ed.

PRE-LAB QUESTIONS:

1. Heating a closed system is generally dangerous. Describe a distillation apparatus that would be a closed system and explain why you should never heat such a system.
2. Boiling chips are usually added before a distillation is started. Explain the purpose of boiling chips in the distillation setup. Should you add boiling chips to a solution that is already heated?
3. Question 3 on pg. 174 in Technique in Organic Chemistry.
4. Would you expect the boiling point of a solution to change if you added a non-volatile solute such as potassium chloride? How? Would an insoluble substance such as sand affect the boiling point?
5. A solution consisting of isobutyl bromide and isobutyl chloride is found to have a refractive index of 1.3852 at 20^{o}C. The refractive indices at 20^{o}C of isobutyl bromide and isobutyl chloride are 1.4368 and 1.3785, respectively. Determine the molar composition (in percent) of the mixture by assuming a linear relation between the refractive index and the molar composition of the mixture.
6. There is a minimum numerical value for the refractive index of a liquid substance. What is this value and why is it the minimum (HINT: take a look at the mathematical formula!)
7. Explain why in Fig 13.16 on page 159 the simple distillation curve starts at a higher temperature and ends at a lower temperature than the fractional distillation. Note both curves are for the same mixture of pentane and hexane.

Safety Information

Add boiling stones before heating liquids. NEVER add boiling stones to a hot liquid!

Never allow a distillation flask to be boiled to dryness. Some compounds, such as ethers, secondary alcohols, and alkenes, can form peroxides through air oxidation, which can cause an explosion if the flask is heated to dryness.

EXPERIMENTAL NOTES:

A: Simple Distillation

In the first part of our experiment we will carry out a simple distillation with a mixture of two liquid compounds. Each group will receive 40 mL of a mixture. Use half for part A and half for Part B. Before you begin the distillation call over your TA and have them check your apparatus!

What You Need to Do Before Lab:

- Complete the reading assignment. Pay special attention to the apparatus in Figure 13.7.
- Rewrite the procedure in section 13.3 ***in your own words***, noting the following changes:
  - Accurately measure the amount of unknown mixture added to your distillation flask using a graduated cylinder. You should use approximately half of the unknown mixture that you are given.
  - Use a 10 mL graduated cylinder to collect the distillate instead of a round bottom flask.
  - Read the temperature for each 0.5 mL of distillate collected.

Data to Record:

- Your observations!
- Total volume of unknown added to distillation flask.
- Temperature reading for each 0.5 mL of distillate collected.
- Construct a distillation curve for the simple distillation (see Figure 13.5 for example).
- Identify the **possible** components of your mixture based on boiling point.
- Determine the percent composition of your unknown mixture.

B. Fractional Distillation

In the second part of the experiment, we will carry out a fractional distillation with the remaining portion of our unknown mixture. Before you begin the distillation call over your TA and have them check your apparatus!

What You Need to Do Before Lab:

- Complete the reading assignment. Pay special attention to the apparatus in Figure 13.17 .
- Rewrite the procedure in section 13.4 ***in your own words***, noting the following changes:
  - Accurately measure the amount of unknown mixture added to your distillation flask using a graduated cylinder. You should use approximately half of the unknown mixture that you are given.
  - Use a 10 mL graduated cylinder to collect the distillate instead of a round bottom flask.
  - Read the temperature for each 0.5 mL of distillate collected.

Data to Record:

- Your observations!
- Total volume of unknown added to distillation flask.
- Temperature reading for each 0.5 mL of distillate collected.
- Construct a distillation curve for the fractional distillation (see figure 13.16 for an example).

- Identify the **possible** components of your mixture based on boiling point.
- Determine the percent composition of your unknown mixture.

C. Identification of Unknown by Refractometry

In the final part of this experiment, we will confirm the identities of our unknowns using the refractive index of the liquids. Note: Refractive indices can be measured to the ten-thousandths place. The digit in the last place, however, can vary widely. We will round our measurements to the thousandths place.

What to Do Before Lab

- Complete the reading assignment!
- Rewrite the procedure in sections 16.3 and 16.4 ***in your own words*** for the following samples:
  - Ethanol
  - Water
  - Sample of distillate from lower boiling component in fractional distillation when collected volume reaches 2 mL
  - Sample of distillate from higher boiling component in fractional distillation when temperature reaches a steady reading

Data to Record

- Your observations!
- Refractive indices of each sample
- Identify **possible** components of your unknown mixture based on refractive index. Refer to the table below.

Table 6.1 Boiling Points and Refractive Indices of Selected Liquids

Compounds	Boiling Points (^oC)	RI ($n D^{20}$) at 20^oC
Acetone	56.5	1.3590
Methanol	64.7	1.3290
Hexane	68.8	1.3750
Ethanol	78.5	1.3600
Cyclohexane	80.7	1.4260
2-Propanol	82.0	1.3728
t-Butyl alcohol	82.2	1.3870

Compounds	Boiling Points (^oC)	RI ($n D^{20}$) at 20^oC
1-Propanol	97.0	1.3840
Heptane	98.4	1.3870
Water	100.0	1.3290
2-Butanol	108.0	1.3960
Toluene	110.0	1.4960
1-Butanol	117.2	1.3990
4-Methylheptane	118.0	1.3840

NOTES FOR WRITING YOUR DISCUSSION:

Your Theory section should include:

- An brief overview of simple and fractional distillation and their utility in separation of liquids
- A discussion of the major differences in the two types of distillation – be specific and detailed. Make sure you cover the following: fractionating column, temperature gradient, theoretical plates!

Your Results section should include:

- A graph that combines the distillation curves you obtained for both simple and fractional distillations (see figure 13.16 for an example)
- A table such as the one below summarizing and organizing all the data you collected

	SIMPLE DISTILLATION			***FRACTIONAL DISTILLATION***			
Fractions	***Volume***	***% Composition***	***Boiling Point***	***Volume***	***% Composition***	***Boiling Point***	***Refractive Index***
1							
2							
3							

Your Discussion section should include:

- An explanation of how you deduced the identities of the two components of your unknown mixture.
- An evaluation of the effectiveness of each type of distillation. Which one was better for your mixture

Your Sources of Error/Future Experiments section should include:

- Discuss whether the total volume of the isolated components of the mixture corresponds to the volume of mixture you started with. If it doesn't propose what might have caused that discrepancy.
- Comment on the reliability of your results and propose <u>different</u> experiments/ techniques that would address the identification and the determination of the purity of the two components of the unknown.