Objectives

- Separate miscible liquid organic compounds by boiling point
- Understand concept and types of distillation

Background

Distillation

Many organic compounds are liquid at room temperature. If a reaction results in a mixture of miscible liquids, the separation of the liquids requires a new technique known as distillation. Distillation uses the different boiling points of liquids in a mixture to separate the components.

Concept of Distillation

Many organic compounds are volatile, which means they have relatively high vapor pressures and low boiling points. Distillation uses this volatility by boiling the liquid mixture in a single flask and condensing the vapors that travel into the apparatus, allowing a liquid to be collected at the other end. As long as there is a difference in boiling points between the liquids in the mixture, one component will distill over before the other.

Pure organic compounds will distill over a very narrow boiling point range. If the boiling point range or distilling range is too wide, the collected compound is likely impure and a complete separation was not obtained. However, not all compounds with narrow boiling point ranges are pure. An azeotrope is a mixture of two liquids which has a constant boiling point and composition throughout distillation. An example azeotrope is a mixture of ethanol and water; ethanol has a boiling point of 78.4 ºC and water has a boiling point of 100 ºC but a mixture of 95% ethanol and 5% water has a boiling point of 78.1 ºC .When distilling ethanol and water, the collected ethanol will be 95% with 5% water due to the azeotrope.

Types of Distillation

There are many types of distillation that differ in the components of the distillation apparatus. All distillation set-ups do have similarities, though. The flask with the mixture is attached to some form of condenser, which empties into a new container. The set-ups also include a thermometer to monitor the vapor temperature compared to the expected boiling points. All set-ups must also be open to a gas environment in some way, whether that is open to air or another gas. A closed distillation system could allow the pressure to build up and cause an explosion.

The type of distillation used in this experiment is simple distillation. The set-up for a simple distillation involves a flask in which to heat the mixture, a connector known as a distillation head, a condenser, and a collection vessel. In large scale simple distillations, the condenser is often jacketed with running water to keep it cool. However, the condenser in a microscale set-up, like that used in this experiment, only uses air to cool the vapor in the condenser, as shown in Figure 1.

Figure 1 Microscale simple distillation set-up with air condenser

Another common type of distillation is fractional distillation, which adds a fractionating column to give better separation of liquids with similar boiling points. The fractionating column is included right above the heated flask, as shown in Figure 2. The column contains extra surfaces on which the high boiling point component can condense back to liquid into the flask, releasing heat that helps vaporize the low boiling point component and move it through the system.

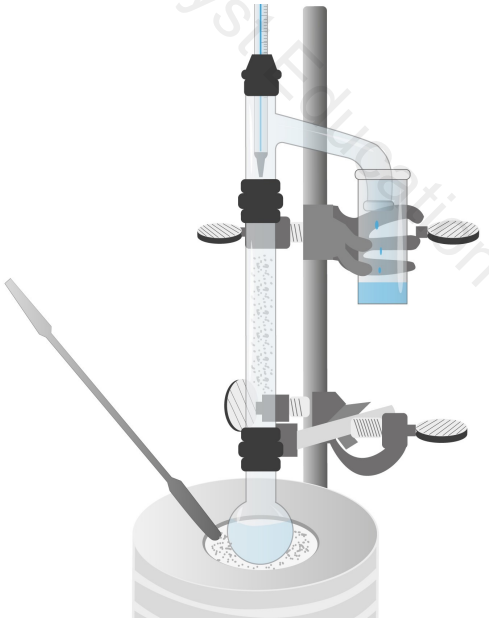

Figure 2 Microscale fractional distillation set-up

Fractional distillation allows for a better separation of liquids with a boiling point difference of 40 degrees or less. Consider the plot in Figure 3 following the distillation of two liquids with boiling points of 81 degrees and 110 degrees.

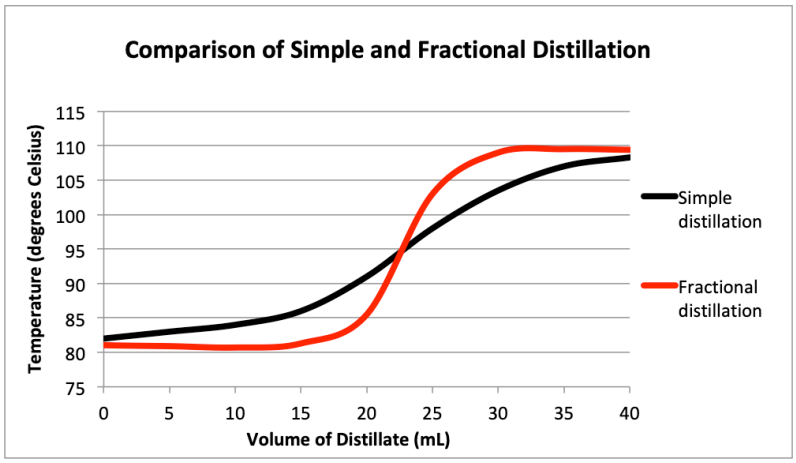

Figure 3 Comparison of simple and fractional distillation temperature behavior over a distillation

There is no sharp transition in the black line representing the temperature behavior during the simple distillation, indicating that there is a wide range of collected distillate containing a mixture of both liquids. The red line has a much sharper transition, which indicates a cleaner separation between the two liquids.

Materials

- Mixture of cyclohexane and toluene
- Microscale glassware kit
- Boiling chips
- Hot plate
- Wet paper towel

Safety goggles are required!

All work should be performed in the fume hood.

Cyclohexane is flammable and will cause skin irritation. Toluene is flammable, toxic, and can cause skin irritation.

Do not boil liquids in a closed system as pressure could increase to create an explosion.

Add boiling chips before heating so that the liquid does not boil over and out of the apparatus.

Procedure

Distillation

1. Place 3 mL of the cyclohexane and toluene mixture in a round bottom flask with a boiling chip.
2. Assemble the distillation apparatus as generally shown in Figure 1, ensuring that the thermometer bulb should be aligned at or slightly below the Y in the distillation head.
3. Turn on the hot plate to start heating the solution. Monitor the temperature and the boiling of the solution throughout the distillation process.
4. If condensation reaches the condenser arm but does not drop into the collection vessel, wrap a wet paper towel around the condenser arm.
5. Record the temperature of the distillate when the first drops are recovered.
6. Adjust the hot plate so that the distillation rate is only two to four drops per minute. Overheating to gain speed will result in an improper distillation and separation.
7. Monitor the temperature closely and note when the temperature begins to rise again.
8. Once the temperature of the vapor rises above 95 ^{o}C, collect the distillate into a different tube as you have changed from one component to the other.
9. Measure the volume of each separate collected component from the distillation.
10. Discard each component of the separated mixture into the non-halogenated waste.
11. Wait for the glassware to cool completely before disassembling the distillation apparatus. Then, clean all the glassware well and put it away.

Pre-Lab Questions

Prepare for lab by completing and understanding the answers to these questions. Refer to the Background or another resource, such as your textbook, if necessary.

1. What is the boiling point of each component of the cyclohexane/toluene mixture used in this experiment?

2. What precautions should you take when setting up a distillation apparatus?

3. What could happen if you overheat the solution in the flask?

4. What is an azeotrope?

5. What is the basis of the separation being performed in this experiment?

Lab 1: Separation by Distillation Report Sheet

Name _____ Section _____

Date _____ Instructor _____

Distillation

Volume of the starting solution (mL) _____

Cyclohexane Component

Vapor temperature when distillation of cyclohexane started _____

Vapor temperature when distillation of cyclohexane finished _____

Volume of cyclohexane collected (mL) _____

Mass of cyclohexane (*d=0.779 g/mL)* collected (g) _____

Space for calculations:

Toluene Component

Vapor temperature when distillation of toluene started _____

Vapor temperature when distillation of toluene finished _____

Volume of toluene collected (mL) _____

Mass of toluene (*d=0.867 g/mL)* collected (g) _____

Space for calculations:

Post-lab Questions

1. What is the percentage of cyclohexane in the mixture?