Distillation Experiment

CHM226

Background

The distillation process is a very important technique used to separate compounds based on their boiling points. A substance will boil only when the vapor pressure of the liquid is equal to the external pressure being applied by the surroundings. Distillations can be used to efficiently purify volatile (i.e. low boiling) compounds. The general concept of distillation involves the boiling of a mixture, resulting in the lower boiling compounds boiling off first. This compound is then collected when the vapors are cooled on a condenser.

Several different techniques exist for specific applications. The most commonly used method is the simple distillation apparatus (Figure 1). This results in the vapors being collected and affords one solution in moderate purity. It is difficult to separate compounds by this method unless they have a large difference in boiling points (>25 °C), or if one is trying to removing a liquid from a solid. A similar apparatus is used during a fractional distillation (Figure 2). The primary difference is that a fractionating column provides significantly more surface area, and essentially results in repetitive simple distillations being formed throughout the column. The consecutive evaporation and condensation allow for the separation and purification of compounds with similar boiling points.

Boiling points are directly proportional to pressure; therefore as the pressure is decreased the boiling point will also decrease. Compounds with very high boiling points can be distilled much easier if a vacuum is applied. Vacuum distillation is a common technique which allows for the distillation of high boiling compounds under mild conditions. Both the simple and fractional distillation apparatus can be performed under vacuum by attaching the vacuum line to the fume hood vented adapter near the collection flask.

The Hickman distillation apparatus is a micro-scale purification apparatus that is used to distill small amounts of materials (Figure 3). This is similar to a simple distillation and only one fraction is usually collected. The purity is again related to the difference in boiling points, and fractionating columns can be employed to increase separation and purity. We will be using this apparatus today to separate a mixture of ο-xylene and cyclohexane.
Figure 1- Simple Distillation Apparatus\textsuperscript{1}
Figure 2- Fractional Distillation Apparatus²
**Procedure:**

Use a graduated cylinder, a pipet bulb, and two disposable glass pipets to combine 2.0 mL of o-xylene and 2.0 mL of cyclohexane in a 5.0 mL conical vial. Record the exact volumes in your notebook. Mix the solutions together using the pipet. Collect 0.5 mL of the mixture in a gas chromatography (GC) vial. Clearly label the vial with your initials and corresponding sample name. Add a boiling chip or stirring vane to the conical vial.

Use the proper size adapter to connect the 5.0 mL conical vial directly to the Hickman still head. Place the conical vial into the sand bath. The sand should be close to or above the liquid volume. Using two clamps secure the apparatus by clamping both the vial and the still head. Make sure the apparatus is vertical and straight. Carefully insert the thermometer into the Hickman still head, having the bulb of the thermometer rest on top of the glass spikes.

Begin heating the mixture using a high setting on the hot plate (6+). If a stirring vane was used begin stirring the mixture. **Record the initial temperature** at which the distillate first starts to collect in the Hickman still head with proper significant figures. Allow the mixture to boil until at least 0.5 mL of distillate has been collected. **Record the maximum temperature** at which the distillate was collected. Transfer 0.5 mL of the distillate to a GC vial using either a glass pipet or by pouring the cool solution out of the Hickman still head into a beaker, and subsequently transfer by pipet to a GC vial.
Give the two clearly labeled GC vials (original mixture, and purified distillate) to your TA for GC analysis. Obtain two standard spectra for the pure o-xylene and cyclohexane from your TA. Before leaving lab you should have four GC spectra: pure o-xylene, pure cyclohexane, original mixture, and purified distillate.

**References**

1.) Apparatus http://www.btinternet.com/~melee3d/revision/chemistry/alcohols.html

2.) Apparatus http://www.chemguide.co.uk/physical/phaseeqia/idealfract.html
**Pre-lab Questions (10pts):**

1. a.) What is the normal boiling point of o-xylene? (1pt)

   b.) What is the normal boiling point of cyclohexane? (1pt)

   c.) Assuming a mixture of equal volumes of o-xylene and cyclohexane, which of these will distill off first? Why? (3pt if explanation is correct, 0pts if not)

2. Consider a large scale mixture of acetone and water (> 1 L):

   a.) Assuming equal volumes, which of these will distill first? (2pt)

   b.) Which **one** apparatus would be most likely used to perform this separation (simple, fractional, vacuum, or Hickman)? Why? (3pt if explanation is correct, 0pts if not)
Post-Lab Questions (10pts total)

1.) What is the retention time of o-xylene in your GC spectra? Label this peak on all spectra containing o-xylene. (1pt)

2.) What is the retention time of cyclohexane? Label this peak on all spectra containing cyclohexane. (1pt)

3.) Consider the synthesis of the solid imine product below. Give two examples of how distillation can be used before or after the reaction. (Hint: Think about physical properties of each compound or solvent in the reaction. Room temperature is ~25 °C) (2pts, 1pt each)

\[
\begin{align*}
\text{H} & \quad \text{O} \\
\text{H} & \quad \text{N} \\
\text{N} & \quad \text{H} \\
\text{N} & \quad \text{H} \\
\text{N} & \quad \text{H}
\end{align*}
\]

\[
\begin{align*}
4\text{-quinolinecarboxaldehyde} & \quad \text{excess aniline} & \quad \text{solid imine-product} \\
\text{m.p.} & = 45-52 ^\circ \text{C} & \text{m.p.} & = -6.3 ^\circ \text{C} \\
& & \text{b.p.} & = 184.13 ^\circ \text{C}
\end{align*}
\]

4.) Name one benefit of using a vacuum distillation apparatus for a high boiling compound instead of a simple distillation apparatus. (2pts)

5.) Name one physical observation indicating that the entire first distilling compound has been collected. (2pts)

6.) Submit all 4 clearly labeled GC s (2pts, 0pts without labels)