Purpose

To explore the differences between simple and fractional distillation on a sample of 20% ethanol/water.

Learning Objectives

Perform a simple distillation of a 20% ethanol/water mixture.

Perform a fractional distillation of a 20% ethanol/water mixture.

Compare the efficacy of simple distillation vs. fractional distillation.

Equipment

- Hotplate
- Heating Mantle
- 25 mL Round-bottom flask
- Two Keck clamps
- Distillation head
- Thermometer
- Rubber septum with large hole
- Water-jacketed condenser
- Graduated cylinder

Chemicals

- Boiling stones
- 20% Ethanol/water

Theory and Background

Distillation is undoubtedly the most important purification technique for organic liquids (bp $< 300\,°C$). It involves heating a liquid to its boiling point at atmospheric or reduced pressure to convert it to a vapor and then condensing the vapor back to a liquid by cooling. The **boiling point** of a liquid is the temperature at which the vapor pressure of the liquid equals the atmospheric or applied pressure. Vapor pressure is related to the strength of the intermolecular forces present in the liquid as well as the temperature of the liquid. It is not related to the exposed surface area or the volume of the space above the liquid (assuming there is not so much space that all of the liquid evaporates before equilibrium can be established). These other factors may influence the rate at which vapor pressure equilibrium is achieved, but the ultimate result will be the same for a given liquid at a given temperature. The amount of vapor pressure generated is thus tied to the energy needed to overcome the forces that hold the liquid together. Vapor pressure is not a linear function of temperature, but instead has a more complicated logarithmic relationship.

A homogeneous mixture (i.e., a solution) of two liquids boils when the vapor pressure of the mixture is equal to the applied pressure, that is, when the sum of the partial pressures of the components (P_A, P_B, P_C ...) equals the applied pressure, P_{total}; thus, at the boiling point [$P_{total} = P_A + P_B + P_C$].

Raoult's Law

All liquids exert a **vapor pressure**: the pressure exerted by a vapor that is at thermodynamic equilibrium with its condensed form. Solids have almost no vapor pressure. Vapor pressure is not linear with temperature but has a complicated logarithmic relationship. At the boiling point for an ideal mixture, the total vapor pressure equals the sum of each individual component's partial pressure [$P_{total} = P_A + P_B + P_C$], and equals the external pressure. Raoult's Law states that the partial pressure of component A (P_A) at any given temperature is equal to the vapor pressure of the pure substance ($P°$) at that temperature, multiplied by its mole fraction in solution (X_A). In other words, [$P_A = P°X_A$ and $P_B = P°X_B$] and so on. For an ideal solution, the vapor is "enriched" in the lower boiling point component when compared with the liquid mixture. If the mixture is non-ideal, Raoult's Law does not hold and an **azeotrope** will form.

As the distillation proceeds, the mixture becomes depleted of the lower boiling component. This analysis is only theoretical and in practice it's a dynamic situation. As soon as the first few drops of distillate are collected from boiling the original mixture, the mixture becomes depleted of the lower boiling component A and its boiling point rises. However, in theory, it is possible to separate a mixture of two liquids into its components by carrying out a series of simple distillations. This can better be achieved using the process of fractional distillation.

Procedure

Part 1. Simple Distillation

> Do not perform this experiment in the hood. The draft will cool your apparatus too much for distillation to occur.

1. Assemble the apparatus for simple distillation as shown in Figure 2A.1. The cap on the distillation head should have a rubber septum with a large hole. The thermometer should be placed through this hole so there is a seal. The top of the thermometer bulb should be directly below the arm of the distillation head.

2. Add 15 mL of a 20% ethanol/water solution to a 25 mL round-bottom flask.

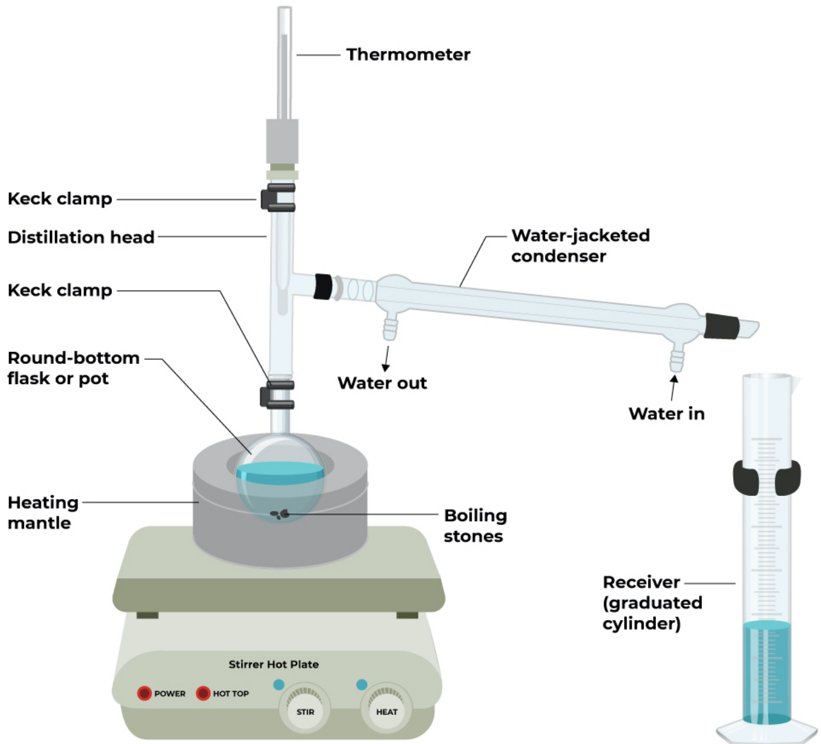

Figure 2A.1: Semi-micro scale simple distillation setup

3. Add 2–3 small boiling stones into the round-bottom flask.

> **Boiling chips** have fissures that enable gases to collect without a violent eruption. This violent eruption is sometimes referred to as "bumping." Adding chips helps the liquid to boil evenly and nonviolently.

4. Place a 10 mL graduated cylinder at the end of the condenser as a receiver.

5. Turn on the water to the condenser and adjust flow to a trickle.

6. Begin heating with the hot plate. Watch the flask to see that it does not boil so vigorously that the liquid solution "bumps" into the distillation head. Distillation should be drop-wise.

7. Record the temperature on the thermometer for every 0.5 mL of distillate that is recovered in the graduated cylinder.

8. Continue the distillation until 8 mL has distilled or the temperature reaches 100 °C (the boiling point of pure water).

9. Stop the distillation by turning off the hot plate and raising the clamp holding the apparatus so that it is above the heating source.

10. In your notebook, make a graph of temperature versus distillation volume.

Part 2: Fractional Distillation

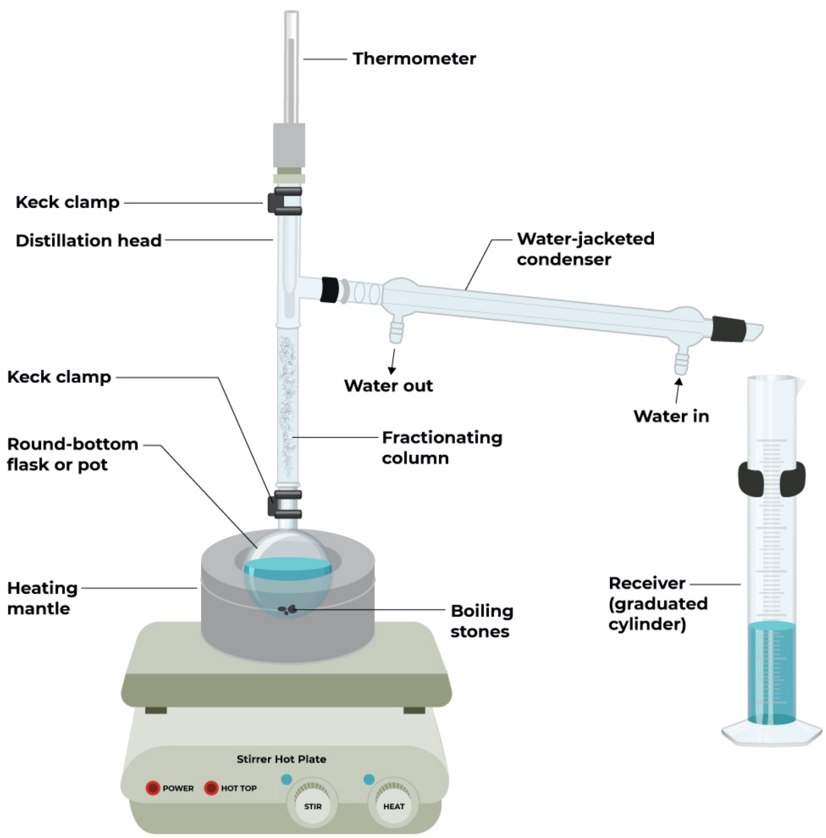

Figure 2A.2: Semi-micro scale fractional distillation setup

1. Once the distillation glassware has sufficiently cooled, install a fractionating column with metal turnings between the round-bottom flask and the distillation head, as shown in Figure 2A.2.

2. Repeat the distillation procedure above using a fresh 15 mL of the 20% ethanol/water mixture. The fractionating column can be loosely covered with aluminum foil.

Discussion

As this is your first lab report, it will be worth you time to review the guidelines and expectations for lab reports that are found in the syllabus and in the document “Tips for Writing a Good Lab Report” (available on Canvas).

In your lab report, include the following topics:

- Construct an original graph of your data and include this with your lab report. You will have two sets of data (one for simple distillation, one for fractional distillation). You should include both sets of data on the same graph.

- Use your data and graph to argue that fractional distillation does or does not produce liquids that are purer than simple distillation

- Does either method result in the distillation of pure ethanol? Indicate any sources of error and how you could limit them

- If fractional distillation does produce liquids that are purer, are there any downsides to using fractional distillation?