Simple and Fractional Distillation

Introduction:
The boiling point of liquid is the temperature at which the vapor pressure of the liquid becomes equal to the pressure of the system or to the atmospheric pressure. When the temperature of a liquid is raised so that its vapor pressure becomes equal to the atmospheric pressure or the pressure of the system, and maintained at that temperature, the liquid will pass into the vapor state. This process is known as distillation. When a chemist speaks of distillation, he or she generally means the combined process of distillation followed by condensation of the vapor into a liquid.

The distillation range can be an indication of the purity of the liquid. Any liquid with a wide boiling or distilling range is impure. In most cases, if the distilling range is narrow and constant, the liquid is pure. If the liquid is impure, yet it has a narrow and constant distilling range, it is known as an azeotrope.

Acetone, CH₃COCH₃, is a colorless, flammable liquid with a boiling point of 56-57 °C and is completely miscible with ethanol (ethyl alcohol). Ethanol, CH₃CH₂OH, has a boiling point of 78-79 °C. In Part A of this experiment, you will do a simple distillation of this mixture, and in Part B a fractional distillation of the same mixture. You will determine the ratio of acetone to ethanol.

Part A: Simple Distillation of an Acetone-Ethanol Mixture

Procedure:
1. Make a table in your lab record book of Temperature, °C vs. Volume, mL.

2. Pour 30 mL of the acetone-ethanol mixture into your 50 mL round bottom flask and add 2 boiling chips. Place the round bottom flask in a heating mantle and clamp the round bottom flask. Set up the simple distillation apparatus shown in Figure 1. This is similar to the one shown in your text (p.181, Figure 12.7, 4th ed). However, you will be using a graduate cylinder as a receiving flask. Be sure to apply grease around the ground glass joints and attach clamps as needed.

3. Start the water running slowly through the bottom of the condenser and have the instructor check the set up before starting to heat the flask. Make sure the heating mantel is plugged into the Variac, not directly to the socket.

4. Regulate the heat control, starting at position 5-6 and decreasing to a lower numerical value on the heat control, so that the rate of distillation is no more than about 1 drop every 2 seconds. Collect the distillate in the graduated cylinder. Record the temperature after every 2 mL of distillate. Distill until you have collected approximately 26 mL. Never distill to dryness.

5. Make a graph, plotting temperature vs. volume of liquid collected and draw a smooth curve through the points.

6. Discard the collected acetone-ethanol mixture into the appropriate waste bottle located in the hood. Do not pour organic liquids into the sink.
Part B: Fractional Distillation of an Acetone-Ethanol Mixture

Procedure:
1. Make a table in your lab record of Temperature, °C vs. Volume, mL.
2. Make a fractionating column by placing a piece of steel wool into a condenser. Do not pack the condenser tightly.
3. Pour another 30 mL of the acetone-ethanol mixture into a 50 mL round bottom flask and add 2 boiling chips. Place the round bottom flask in a heating mantle and clamp the round bottom flask. Set up a fractional distillation apparatus as shown in the Figure 2. This is similar to the one shown in your text (p. 191, Figure 12.17, 4th ed). However, you will be using a graduate cylinder as a receiving flask. Be sure to apply grease around the ground glass joints and attach clamps as needed.
4. Start the water running slowly through the bottom of the condenser and have the instructor check the set up before starting to heat the flask. Make sure the heating mantel is plugged into the Variac, not directly to the socket.
5. Regulate the heat control, starting at 7-8 and decreasing to a lower numerical value on the heat control unit, so that the rate of distillation can be maintained at less than 20 drops per minute. Collect the distillate in the graduated cylinder. Record the temperature after every 2 mL of distillate. When the temperature starts to drop as shown on your thermometer, you will have to turn up your heat control so that more heat is supplied to distill the higher boiling fraction. Distill until you have collected approximately 26 mL. Do not distill to dryness.
6. On the same graph as the simple distillation, plot temperature vs. volume of liquid collected using a different colored pen or different marks (circles, triangles, +...) to differentiate between the points used for the simple distillation graph. Draw a smooth curve through the markings.
7. Discard the collected acetone-ethanol mixture into the appropriate waste bottle located in the hood.

Questions
1. Why is it necessary to add boiling chips to the round flask?
2. Considering your results, which method is better for separating acetone from ethanol, simple or fractional distillation? Justify your answer.
3. From your graph, what is the percent volume of acetone in the original mixture?
4. What is the purpose of the condenser?
Also, answers questions 3 and 4 on page 205, 4th ed (p. 174, 3rd ed) in TOC.

Conclusion: Summarize your results and cite any important considerations that should be observed when performing a distillation.
Figure 1 Simple Distillation Setup
(Similar to Fig. 12.7, p 181 in your lab book except a graduate cylinder is used instead of round bottom flask for the receiving flask.)

Figure 2 Fractional Distillation Setup
(Similar to Fig. 12.17, p 191 in your lab book except a graduate cylinder is used as the receiving flask.)