SIMPLE AND FRACTIONAL DISTILLATION

Read technique 11 from the start of the chapter up to and including section 11.5 – Azeotropic Distillation)

Introduction

The boiling point of a liquid is the temperature at which the vapor pressure of the liquid becomes equal to the pressure of the atmosphere above the liquid. Some compounds are distilled "under reduced pressure", or "under vacuum". This means that the distillation is carried out in an apparatus in which the pressure has been reduced. What will this do to the boiling point of the liquid in the apparatus? Why might chemists need to run distillations "under reduced pressure? There is a useful chart at the back of the Aldrich catalog which you can use to determine the boiling point of a liquid at any pressure, if you know the boiling point at atmospheric pressure.

When the temperature of a liquid is raised, the vapor pressure exerted by that liquid increases. When the vapor pressure becomes equal to the pressure of the atmosphere above the liquid, the liquid will pass into the vapor state (it will "boil"). If you then cool down the vapors, they will condense and reform the liquid. Most chemists use the term "distillation" to refer to the combined process of heating a liquid until it boils and passes into the vapor state, followed by condensation of that vapor into a liquid.

The distillation range of temperature can be an indication of the purity of the liquid. A liquid with a wide boiling or distilling range is impure (similar to the melting point). Distillation is also a valuable technique that is used quite often in the chemistry laboratory. If you have a mixture of two liquid compounds, both compounds will contribute to the vapor pressure of the total mixture. But the composition of the vapors is not the same as the composition of the liquid. There is a greater percentage of the lower boiling compound in the mixture of vapors than there is in the mixture of liquids. Therefore, if we boil a mixture of two liquids and condense the vapors, the condensed (distilled) mixture will have a greater percentage of the lower boiling liquid than the original mixture did. This is because it takes less energy for the lower boiling liquid to enter the vapor phase. This is shown graphically in a temperature/composition curve (in section 11.2). You should make sure that you know how to use this curve. This graph differs for every mixture of liquids. The big question today is: How big a difference in boiling points do you need to be able to completely separate and purify a mixture of liquids? Well, that's what this lab is designed to demonstrate! Your lab instructor will discuss this principle in more detail in lab. You should make sure that you understand the theory of distillation and the definition of a theoretical plate.

If the distilling range is narrow and constant during a distillation, it usually means that the liquid is pure. However, some liquids will form an azeotrope when they are combined. An azeotrope is a mixture of liquids that has a narrow constant boiling range. One example of an azeotrope is the mixture of ethanol (alcohol) and benzene (a carcinogenic substance). If you distill a mixture of these liquids, you will not be able to separate them completely. This is because they will form an azeotrope, and both compounds will always be present in the vapor stage until one or both of the compounds has been completely distilled. All of the ethanol we use in this class will have some benzene (a small percentage) mixed in with it. Which means that it is not safe to drink, and if you distill it, it still will not be safe to drink because there will still be benzene mixed in with the ethanol! There are many other azeotropes. However, we will only be using mixtures that

exhibit typical distillation properties. Today we will be distilling a mixture of acetone and ethanol. The two liquids are completely miscible (they will mix together), and the boiling point of acetone is
56-57 ˚C. The boiling point of ethanol is 80 ˚C.

Experimental Procedure: (we will do this experiment with partners, one does simple and one does fractional distillation)

Simple Distillation

Make a table in your lab record book of Temperature (˚C) vs. Volume (mL).

Pour 30 mL of the acetone-ethanol mixture into your 50 mL round bottom flask and add two boiling chips. Place the flask in a heating mantle (do not turn on the heating mantle at this point) and clamp the flask. Then assemble the apparatus for a simple distillation as shown at the end of this handout. There will also be a demonstration of this set-up in the lab.

Start the water running slowly (you only need a trickle of water coming out of the rubber tubing) through the condenser and have your instructor check the set-up before turning on the heating mantle. After your instructor has checked your set-up, turn on the heating mantle, starting at position 9. You will have to soon decrease the setting on the heating mantle in order to form **only one drop** of distillate per second. When the temperature of your thermometer starts to drop, you may have to turn your heat control back up to 9 in order to distill the higher boiling fractions.

Record the temperature after every 2 mL of distillate is collected in the graduate cylinder. Never distill to dryness. You should leave about 3-5 mL of liquid behind in the round bottom flask. Discard the collected acetone-ethanol mixture into the appropriate waste bottle in the hood. **DO NOT POUR ORGANIC LIQUIDS INTO THE SINK!** (let us tread lightly upon the earth!) Make a graph, plotting temperature vs. volume of liquid collected; and draw a smooth curve through the points. You can use this same graph to plot your fractional distillation data.

Fractional Distillation

Make a table in your lab record book of Temperature (˚C) vs. Volume (mL).

Pour 30 mL of the acetone-ethanol mixture into your 50 mL round bottom flask and add two boiling chips. Place the flask in a heating mantle (do not turn on the heating mantle at this point) and clamp the flask. Then assemble the apparatus for a fractional distillation as shown at the end of this handout. We will use a condenser loosely packed with steel wool as our fractionating column. There will also be a demonstration of this set-up in the lab.

Start the water running slowly (you only need a trickle of water coming out of the rubber tubing) through the condenser and have your instructor check the set-up before turning on the heating mantle. After your instructor has checked your set-up, turn on the heating mantle, starting at position 9. You will have to soon decrease the setting on the heating mantle in order to form **only one drop** of distillate per second. When the temperature of your thermometer starts to drop, you may have to turn your heat control back up to 9 in order to distill the higher boiling fractions.

Record the temperature after every 2 mL of distillate is collected in the graduate cylinder. Never distill to dryness. You should leave about 3-5 mL of liquid behind in the round bottom flask.

Discard the collected acetone-ethanol mixture into the appropriate waste bottle in the hood. **DO NOT POUR ORGANIC LIQUIDS INTO THE SINK!** (let us tread lightly upon the earth!)

On the same graph as the simple distillation, again plot the temperature vs. volume of liquid collected; and draw a smooth curve (use a different colored pen) through the points. You can use this same graph to plot your fractional distillation data.

Questions

1. What is the purpose of the boiling chips?

2. Using the boiling point-composition curves for a mixture of hexane and pentane in "technique 11" in your lab text (page 132 in the second edition) to answer the following questions.

    a) At what temperature will an 80/20 mixture of hexane/pentane begin to boil?

    b) What would be the composition of the distillate if you did a simple distillation (one theoretical plate)?

    c) If you had a fractionating column that had 2 theoretical plates, what would be the composition of the distillate from this 80/20 mixture? 3 theoretical plates? How good is good enough?

3. Using your results from lab, what is the volume percent of acetone in the mixture? Which data/graph are you using to determine this? Why did you use those results rather than the results from the other lab partner?