Distillation Lab

**Introduction** - Since organic compounds do not usually occur in pure condition in nature, and are accompanied by impurities when synthesized, the purification of materials forms an important part of laboratory work in chemistry. Four general separation procedures are used frequently in organic work in the laboratory and in industry: distillation, chromatography, crystallization, and extraction. The process used in any particular case depends upon the characteristics of the substance to be purified and the impurities to be removed. In order to select the most appropriate process and to employ it effectively it is important that the student understand the principals involved as well as the correct methods of manipulation. Simple distillation is one of the most commonly used purification methods.

If one compound is much more volatile than the other, the compounds can be separated in one vaporization step. Such a step is called simple distillation and uses an apparatus that consists of only a pot, a distilling head, a condenser, and adapter, and a receiver (see Figure 1). When the boiling points of two compounds differ by less than 40ºC, they cannot be separated by simple distillation. Fractional distillation, a process that has the effect of many simple distillations, must be used. A fractional distillation apparatus includes a fractionating column placed between the pot and the distilling head (see Figure 2). Typically, any one of a variety of materials, including glass beads and metal sponge, fill the fractionating column.

![Figure 1. Simple Distillation Apparatus](image1)

![Figure 2. Fractional Distillation Apparatus](image2)

The **boiling point** of a liquid is defined as the temperature at which the equilibrium vapor
pressure of the liquid equals the existing atmospheric pressure. The atmospheric pressure measured in mmHg is the current atmospheric pressure in the laboratory.

The **normal boiling point** is the temperature at which the vapor pressure of a liquid equal 760 mmHg. This is the atmospheric pressure at sea level. This is a reference value. Generally boiling points go down as the atmospheric pressure decreases, or the pressure above them in their container.

The boiling point of a liquid is a physical property of that substance and is used to help identify it, or to determine its purity. Measurement of boiling point can be done in a number of ways. One tactic that may be used to purify a liquid is to **distill** it. The process is common and one that is often used in the chemical laboratory. This exercise will distill a single liquid, and a mixture of liquids. The procedure that will be employed is called **simple distillation**.

Boiling points for a large number of liquids can be found in the *Handbook of Chemistry and Physics*. This reference book is available in the laboratory and the library. Boiling points are often listed with a superscript that indicates pressure. If no reference is given, it is assumed to be at 760 mm Hg.

**Simple Distillation:** A simple distillation apparatus is shown in Figure 1. This consists of a round-bottomed flask connected by means of a distillation adapter to a water-cooled condenser. A thermometer is held in place in the vertical arm of the distillation adapter by a special rubber connector at a height adjusted so that the top of the thermometer bulb is 5 – 10 mm below the opening of the side-arm. A (vacuum) adapter is connected to the lower end of the condenser.

The distilled liquid is collected in a clean, dry receiver, commonly an Erlenmeyer flask or small-mouthed bottle. To reduce vapor losses and minimize fire hazards, it is desirable to insert the lower end of the adapter well into the mouth of the receiver. A distilling assembly must have an opening to the atmosphere to avoid developing a dangerously high pressure within the system when heat is applied.

**Key Point:** When conducting a distillation, the vapor should be richer in the lower boiling component than what you started with.

**Fractional Distillation:** The vapors generated in the pot rise up the fractionating column and encounter cooler surfaces, upon which they condense (see Figure 3).
The condensed liquid is then reheated by rising hot vapors and revaporizes. This process of condensation and revaporization, shown graphically in Figure 5a and 5b, may occur again and again as the vapors rise up the column.

Figure 5  Each condensation and revaporization increases the concentration of the more volatile compound.
Figure 5b Fractional Distillation Phase Diagram. The arrows indicate a theoretical plate.

Each vaporization is represented by a horizontal line connecting the liquid composition curve to the vapor composition curve. Each condensation is represented by a vertical line connecting the vapor curve to the liquid curve. For example, the 50:50 liquid mixture (A) vaporizes to produce a 30:70 vapor mixture (A’). The 30:70 vapor mixture condenses to a 30:70 liquid mixture (B). The 30:70 liquid mixture, in turn, vaporizes to produce a 15:85 vapor mixture (B’), and so on. Each condensation-revaporization results in an increase in the concentration of the more volatile compound. These composition changes are reflected by a decrease in boiling temperature as the mixture moves up the fractionating column. If the condensation-revaporization is repeated a sufficient number of times, the vapors of the more volatile compound reach the top of the fractionating column in a pure form. As these vapors move into the condenser, the compound condenses and is collected as a liquid.

At the same time, the less volatile compound is enriched in the opposite direction. As the condensed liquid falls toward the pot, the pot gradually contains a higher and higher percent of the less volatile compound. Thus a separation of the two compounds is achieved.

Each condensation and revaporation that occurs on a fractionating column is called a theoretical plate. A fractionating column with a large number of theoretical plates accomplishes many condensation-revaporization steps and very efficiently separates the compounds in a mixture.

The fractionating column MUST be positioned vertically so that condensed liquid can percolate down through the rising hot vapors. This percolation promotes equilibrium between the liquid and vapor phases, a condition that allows the column to operate at maximum efficiency and provide an optimum separation. An equally important factor affecting separation of the compounds is the distillation rate. If the distillation is conducted too rapidly, liquid-vapor
equilibria will not be established in the fractionating column, and poor separation of the compounds will result.

As the liquid boils, a condensation line of vapor can be observed as it moves up the distilling head. Once these vapors reach the thermometer bulb, a dramatic temperature increase is observed. The temperature of the vapors in the distilling head provides information regarding the progress of the distillation. Initially, the vapors are rich in the more volatile compound, and the observed temperature is close to the boiling point of that compound. In a distillation with an efficient separation, the initial temperature remains relatively constant until all of that compound is collected. After the compound with the lower boiling point is completely distilled, the temperature rises sharply as the vapors of the higher-boiling compound reach the thermometer bulb. At this time, the boiling point of the higher-boiling compound is observed as it distills into the receiver.

When no fractionating column is used, or when the fractionating column is inefficient, mixtures of the distilled compounds are incompletely separated. This inefficiency is indicated by a very gradual increase in the temperature measure during the distillation. Samples collected at temperatures between the boiling points of the two compounds will consist of mixtures of the two compounds. A comparison of the results of simple and fractional distillation is shown in Figure 6.

![Distillation curves for simple and fractional distillation](image)

**Figure 6** Distillation curves for simple and fractional distillation

When will simple distillation do a reasonable job of separating a mixture?

1. When the difference in boiling points is over 100°C
2. When there is a fairly small amount of impurity, say less than 10%.
3. When one of the components will not distil because of a lack of volatility (i.e. sugar dissolved in water).
Simple Distillation

PROCEDURE

REPORT AND DATA COLLECTION - The collection and graphing of data will be done on a spreadsheet. Download the Excel file to collect your data. No formal lab report is required for this experiment.

EXERCISE 1.1 Distillation of a Pure Compound by Simple Distillation
Assemble the simple distillation apparatus. Run cooling water through the condenser in at the "bottom" and out at the "top". Support the round bottom with a securely applied clamp, use a small heating mantle for heat and collect the distillate in a 25 ml graduated cylinder.

Correct Thermometer Placement:
In the dry round bottom flask add 25 mL of methanol (containing a red dye). Add one or two tiny boiling chips, attach the boiling flask, and make certain that all connections are tight. Arrange a graduated cylinder to serve as the receiver. Heat the flask gently to boiling, and record the temperature when the first drops of distillate collect in the condenser. As the liquid boils, watch for the condensation line of vapor as it moves up the distilling head. To observe and record an accurate temperature reading, the entire thermometer bulb must be immersed in vapor. Adjust the heater to produce distillate at a rate that is no greater than one drop per second.

Continue to distill the liquid slowly (not over 2 mL per minute) and record the distilling temperature at regular intervals during the distillation. Discontinue the distillation (and turn off the heat) when all but 2-3 mL of the liquid has distilled. Record the temperature range from the beginning to the end of the distillation; this is the observed boiling point. Note in the spreadsheet if the boiling point differs from the literature value. Allow the pot to cool for a few minutes, then turn off the water to the condenser.

EXERCISE 1.2 Simple Distillation of a Mixture

Use the same set up as in exercise 1.1. Prepare a mixture of acetone and toluene that is 50% acetone and 50% toluene (mix 15 mL acetone and 15 mL toluene). Collect similar data as in the first exercise (collect approx. 20 mL or more).

EXERCISE 1.3 Fractional Distillation Questions to Answer:

1. The blue line on the diagram illustrates the effect of using fractional distillation to separate a mixture of hexane (C₆H₁₄) and heptane (C₇H₁₆). If one starts a fractional distillation with a 0.20 mole fraction of hexane, what is the approximate mole fraction of hexane in the vapor phase after two evaporation-condensation cycles?

2. How many theoretical plates are required to produce a solution with a mole fraction of hexane greater than 0.90?

3. What is the mass percent of hexane in a mixture with heptane if the mole fraction of hexane is 0.20?