Chapter 16: Distillation

Distillation is the process of heating a liquid until it boils, capturing and cooling the resultant hot vapors, and collecting the condensed liquid. Mankind has applied the principles of distillation for thousands of years. Distillation was probably first used by ancient Arab chemists to isolate perfumes. Vessels with a trough on the rim to collect distillate, called diqarus, date back to 3500 BC. In the Middle Ages and the Renaissance, alchemists developed distillation equipment known as retorts. In the 1800s, distilleries producing brandy, whisky, rum, gin, and vodka were established in Europe and America. The word "alcohol" derives from the Arabic "al-koh'l," translated as "finely divided spirit." Most of us are familiar with pictures of the moonshiner's still: a large boiling pot with long coils of metal tubing used for condensing the alcohol vapors into moonshine, or illegal whisky.

In the modern organic chemistry laboratory, distillation is a powerful tool, both for the identification and the purification of organic compounds. The boiling point of a compound – determined by distillation – is well-defined and thus is one of the physical properties of a compound by which it is identified. Distillation is used to purify a compound by separating it from a nonvolatile or less-volatile material. When different compounds in a mixture have different boiling points, they separate into individual components when the mixture is carefully distilled.

16.1 Compound Identification

A. Boiling Point and Pressure

The boiling point is the temperature at which the vapor pressure of the liquid phase of a compound equals the external pressure acting on the surface of the liquid. The external pressure is usually the atmospheric pressure. For instance, consider a liquid heated in an open flask. The vapor pressure of the liquid will increase as the temperature of the liquid increases, and when the vapor pressure equals the atmospheric pressure, the liquid will boil. Different compounds boil at different temperatures because each has a different, characteristic vapor pressure: compounds with higher vapor pressures will boil at lower temperatures.

Consider a container of water that is very cold. Assume that you are at sea level, where the atmospheric pressure is 760 mm Hg. At its freezing point of 0°C, water has a vapor pressure of 4.6 torr. As you heat the cool water, it becomes hot and its vapor pressure increases. At 100°C, the vapor pressure of the water will equal 760 mm Hg and at this point the water will boil. The normal or standard boiling point of water is 100°C because it is the boiling point at 760 mm Hg which is designated standard atmospheric pressure (also equal to 760 torr or 1 atm). Values reported in the literature are measured at standard atmospheric pressure unless otherwise specified.

The boiling point of a compound is a defined value and therefore is a physical characteristic by which it can be identified, much like the melting point. Unlike the melting point, however, the boiling point is very sensitive to the atmospheric pressure, and therefore it is less dependable as a means of identification. The atmospheric pressure differs with changes in altitude, and it changes from day to day at any one location. If you are in Boulder, Colorado, the atmospheric pressure will always be less than standard atmospheric pressure, and therefore the observed boiling points will always be lower than those reported in the literature. (Because if the external pressure is less than 760 torr, the vapor pressure of any compound will equal the external pressure at a lower temperature.) Therefore, you will need to adjust the observed value to a corrected value before comparing the observed boiling points with literature values.

As a quick "rule of thumb," you can assume that the boiling point drops about 0.5°C for a 10 mm decrease in pressure when around 760 mm Hg. Applying this rule of thumb to the boiling point of water in Colorado, where the barometric pressure usually hovers around 630 mm, water will boil at 93.5°C in Boulder. (760 – 630 = 130, which correlates to 13 × 10 mm decreases, and 13 × 0.5° = 6.5°, 100° – 6.5° = 93.5°.)

A more accurate way to estimate the corrected boiling point is expressed in the following formula:

$$T_{corr} = T_{obs} + 0.00010(760 – p)(T_{obs} + 273)$$

where

T_{corr} is the corrected boiling point

T_{obs} is the observed boiling point

p is the barometric pressure

Example Problem 1

You observe a boiling point of 55°C at 650 mm Hg. What is the boiling point of this compound at sea level?

$T_{corr} = 55 + 0.00010 \ (760 – 650)(55 + 273)$

$T_{corr} = 55 + 3.6 = 58.6°C$

Example Problem 2

What temperature will water boil at when the pressure is 630 mm Hg?

This time, we know T_{corr} and p, but not T_{obs}.

$100 = T_{obs} + 0.00010(760 – 630)(T_{obs} + 273)$

$100 = T_{obs} + 0.013Tobs + 3.55$

$T_{obs} = 95.2°C$

Compare this with the "rule of thumb" calculation of 93.5°C, keeping in mind that "rule of thumb" boiling point corrections are only estimates.

B. Measurement of Boiling Point

Boiling points are usually measured by recording the boiling point (or range) on a thermometer while performing a simple distillation. This method is used whenever there is enough of the compound to perform a distillation.

The distillation method of boiling point determination measures the temperature of the vapors above the liquid. Since these vapors are in equilibrium with the boiling liquid, they are the same temperature as the boiling liquid. The vapor temperature rather than the pot temperature is measured because if you put a thermometer actually in the boiling liquid mixture, the temperature reading would likely be higher than that of the vapors. This is because the liquid can be superheated or contaminated with other substances, and therefore its temperature is not an accurate measurement of the boiling temperature.

16.2 Compound Purification

A. Simple Distillation

Simple distillations are used to purify liquids in the following circumstances:

- The liquid is already almost pure (no more than 10% liquid contaminants)
- The liquid has a nonvolatile component, for example, a solid contaminant
- The liquid is contaminated with a liquid whose boiling point differs by at least 70°C

In the teaching laboratories, simple distillations are the most frequently used method of distillation. "Simple" distillation may be a misleading term to the beginning organic chemistry student, since it takes a lot of practice in simple distillation to become proficient in this technique. It is especially important to do a perfect simple distillation when determining a boiling point for identification purposes.

A simple distillation apparatus is shown in Figure 16-1. The liquid to be distilled is placed in a round-bottom flask (often referred to as the "distillation pot"). The liquid is boiled and the vapors rise up into the still head (also referred to as the Y-adapter) and go down the sidearm into the condenser. The thermometer (held on with a thermometer adapter) is positioned near the side arm of the still head so that it monitors the temperature of the vapors. The vapors condense in the condenser and drip down into the receiving flask.
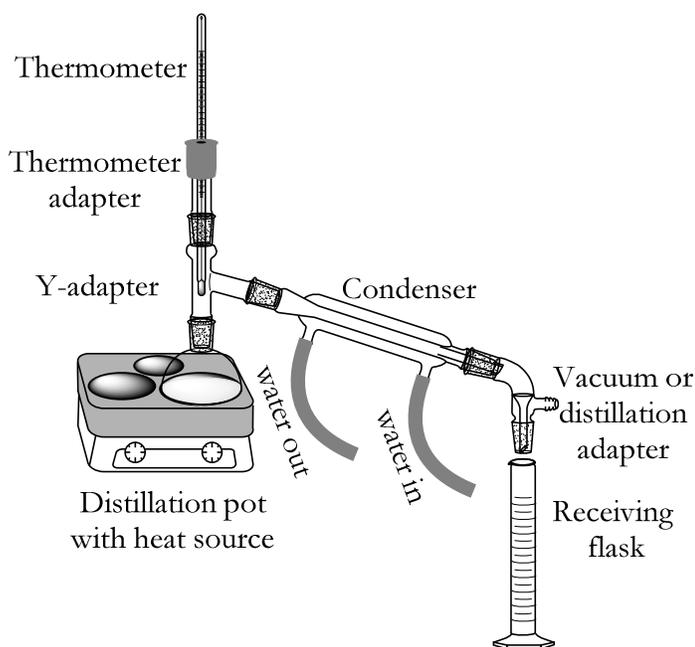

Figure 16-1: The setup for simple distillation.

At the beginning of the distillation, the distillation pot should be between one-half and two-thirds full. If the pot is too full, the surface area is too small for rapid evaporation and the distillation proceeds very slowly. If the pot is not full enough, there will be a large holdup volume and loss of sample. Holdup volume is the amount of vapor in the flask and head along with the liquid required to wet the inner walls of the apparatus. A typical holdup volume is one or two milliliters, which can lead to significant product loss, especially in a small scale experiment.

As the liquid boils, the temperature rises readily to the boiling temperature of the liquid and remains at that temperature until all of the liquid has distilled. The liquid and/or solid that remains in the pot at the end of distillation is called the pot residue; the condensed liquid is called the distillate.

Figure 16-2 shows the temperature versus volume curves for two successful distillations. The graph on the left illustrates the distillation of a liquid that is pure before the distillation begins or is contaminated with a nonvolatile impurity, such as a solid. The temperature rises immediately to the boiling temperature and remains at that temperature until the liquid is distilled. The graph on the right illustrates the distillation of two ideal, miscible liquids with widely differing boiling points. The temperature rises immediately to the boiling point of the lower boiling component and remains at that temperature until all of the lower boiling compound is distilled, then the temperature rises rapidly to the temperature of the higher boiling compound and remains at that temperature until the higher boiling compound is distilled.
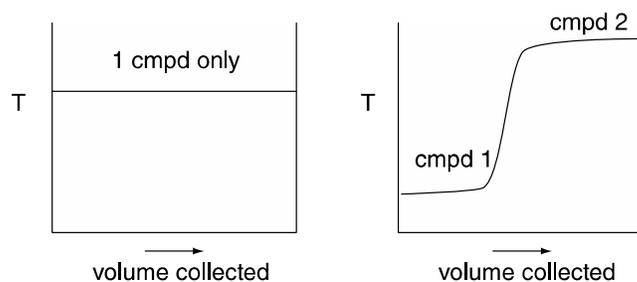

Figure 16-2: Temperature versus volume curves for successful distillations.

The procedure for performing a simple distillation is as follows:

1. Gather your glassware.

Pull all of the glassware you will need from your drawer (see Figure 16-1) and check each item for cleanliness and star cracks. If the procedure you are using does not specify a size of round-bottom flask to use, you should choose a flask based on the amount of liquid you are distilling. The flask should be filled to one-half to two-thirds of its volume.

2. Assemble the apparatus.

Lightly grease each glass-on-glass joint before assembling the apparatus as shown in Figure 16-1, except for the thermometer and thermometer adapter (these will be added later). Hold each joint together with a yellow plastic Keck clip, and clamp the round-bottom flask and the condenser to ring stands. Place the heat source (a stirring hotplate) under the round-bottom flask. If you place the heat source on a lab jack, you will be able to lower it if you need to cool the reaction quickly. Connect two water hoses to the condenser. Water should always flow in through the bottom and out through the top.

3. Fill the distilling flask.

Place a stemmed funnel in the top of the Y-adapter and add the liquid to be distilled (Figure 16-3). Also add a couple of boiling chips – these provide surfaces where bubbles of vapor can form, and prevent sudden eruptions of liquid from the flask. Alternatively, you can fill the round-bottom flask by removing it from the apparatus and filling it directly.
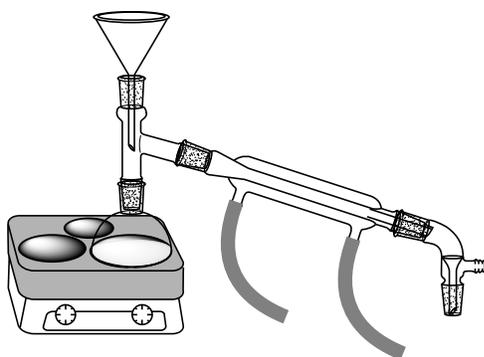
Figure 16-3: Filling the distillation pot for a simple distillation.

4. Add the thermometer.

Now you can add the thermometer and thermometer adapter. Position the thermometer bulb so that it is level with or slightly below the junction in the Y-adapter. It is good practice to add these items last, because the thermometer is the most vulnerable to breakage if your apparatus tips over.

5. Start distilling.

Turn on the water to the condenser to cool it down. Begin heating the distillation pot so that it boils smoothly and the distillate drips into the receiver at about 1–2 drops per second. Do not heat it too fast, or bumping (violent eruptions of large bubbles of liquid into the Y-adaptor) will occur. Bumping can cause impure liquid to be forced through the Y-adaptor and into the distilling pot.

It is not unusual for organic compounds isolated in the lab to have impurities; therefore you might find that the initial temperature at which distillate collects is lower than you expect. This lower boiling distillate is called the forerun. Watch the thermometer, and when the temperature rises to the boiling point of the expected distillate, replace the receiving flask with another flask. Depending on the experiment, you may collect several fractions of distillate at different boiling ranges.

Always discontinue boiling before the distillation pot goes to dryness. Peroxides, which are explosive at higher temperatures, might be present. Discontinue the distillation if the liquid smokes.

One method to avoid boiling to dryness and to increase yield of purified liquid is to add a distillation chaser. With every distillation apparatus, there is a holdup volume that can never be fully distilled over. If you add a high-boiling liquid to the distillation pot, the vapors of this liquid and not the desired lower-boiling liquid will fill the distillation apparatus, effectively "chasing" vapors of the desired liquid through the condenser and into the collection flask. The chaser solvent should boil at a temperature a lot higher than the desired solvent, or else you will have to monitor the last samples collected carefully to avoid contamination of the product with the chaser. The chaser solvent must not form an azeotrope with the desired solvent; use at least enough of the chaser solvent to fill the holdup volume of the apparatus.

Common Distillation Mistakes of the Beginning Student

- Boiling chips are not added to the distillation flask. This causes violent boiling and impurities are bumped into the distillate.
- The thermometer is not placed correctly in the Y-adapter. This causes the measured vapor temperature to be incorrect.

- One or more of the standard taper joints are loose. If they are loose, vapors will escape into the atmosphere instead of condensing in the receiving flask.
- The condenser hoses are loose. This causes you or your neighbor to become quite wet.
- Clamps are not used to secure the glassware. This causes glassware to fall and break; this not only means product is lost, but it means you will have to pay for the broken glassware.
- The mixture is heated too fast. This causes superheated and impure liquid to be forced into the condenser, contaminating the distillate.

B. Fractional Distillation

Fractional distillation is sometimes necessary because there are many circumstances under which simple distillation will not work.

Suppose you mix two compounds, A and B, in approximately equal amounts in a distillation flask. The boiling point of A (TA) differs from that of B (TB) by less than 100°. Observation of the temperature during a simple distillation of this mixture would show a gradual increase in temperature from the boiling point of the lower boiling component A to that of the higher boiling component B (Figure 16-4). If you collected samples (or "fractions") of the distillate as the distillation proceeds, you would note that the first fractions are richer in component A, and the last fractions are richer in component B. The simple distillation will not clearly separate the two components.
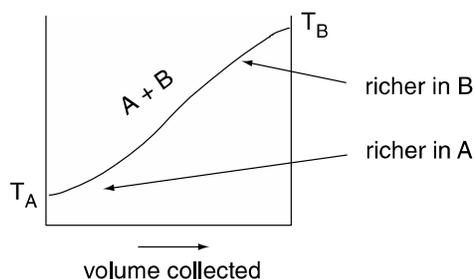

Figure 16-4: Simple distillation curve.

To understand why clear separation of components is not possible in this situation, recall the vapor pressure concepts learned in general chemistry. In an ideal solution of two infinitely miscible liquids, each component contributes to the total vapor pressure at the surface. This principle is stated mathematically in Raoult's law:

$$P_A = X_A P°_A$$

where

P_A is the partial pressure of A

X_A is the mole fraction of A

$P°_A$ is the vapor pressure of pure A

In a mixture of two components, the contribution of each to the total vapor pressure is in direct proportion to the mole fraction of the component. Dalton's law of partial pressures states that the total vapor pressure equals the sum of the individual vapor pressures.

$$P_{total} = X_A P°_A + X_B P°_B$$

Raoult's and Dalton's laws state that the vapor composition above a liquid mixture is dependent both on the vapor pressures of the pure components and on their mole fraction. The higher the vapor pressure of a liquid (which is equivalent to saying "the lower the boiling point of a liquid"), the more vapors of this component will be above the liquid. And, the greater the concentration of a liquid component, the greater the concentration of its vapors above the liquid.

Distillation is a dynamic process, with the vapors continuously condensing into the collection flask, thus changing the mole fractions of the components in the distillation pot. Thus, the composition of vapors and the observed temperature of vapors is constantly changing, as illustrated in Figure 7.3.

Another way to visualize the boiling characteristics of a liquid mixture of components with similar boiling points is in Figure 16-5. This graph illustrates the mole fraction compositions of liquid and vapor phases of a distillation of two liquids at various temperatures. The upper line gives the mole fraction composition of the vapor, and the lower line gives the mole fraction composition of the liquid. If only A is present (mole fraction B = 0), the "mixture" will boil at the boiling point of A, 70°C. Consider a mixture of 0.5 mole fraction B. To determine the composition of the vapors of this mixture, draw a line up to the bottom (liquid) curve, then go horizontally to the vapor curve to point b. Reading from the graph, the mole fraction of B vapors at point b is about 0.15. This means that although the composition of the liquid is half B and half A, the composition of the vapors is 0.15 B and 0.85 A. (This will be the composition of the distillate, too.) Read on across horizontally to determine the boiling point of the mixture at this point: 85°C.
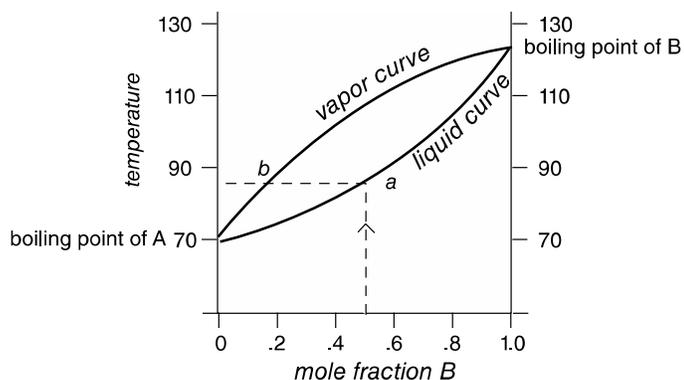

Figure 16-5: Mole fraction of a mixture at different temperatures.

As a result of all this, mixtures of liquids whose boiling points are too similar (separated by less than 70°C) cannot be separated by a single simple distillation. It is possible to separate the compounds if aliquots of the distillate are collected and distilled again; by using a series of traditional simple distillations, the most volatile compound can eventually be purified. However, this process requires many tedious distillations and is quite time consuming.
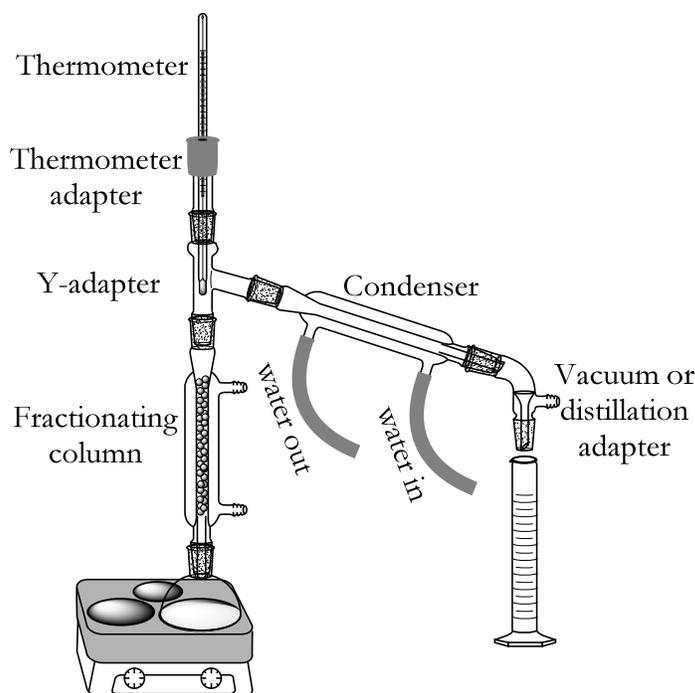
Figure 16-6: Apparatus for fractional distillation.

A much easier procedure is to perform a fractional distillation. A fractional distillation apparatus allows the chemist to perform repeated distillations in one step. To change a simple distillation apparatus into a fraction distillation apparatus, fractionating column is inserted between the boiling flask and the Y-adapter (Figure 16-6). The fractionating column consists of a column packed with glass beads, metal or glass helices, or a specially constructed Vigreux column. The important component of the fractionating column is a very large surface area.

In fractional distillation, a series of simple distillations occur on the surfaces of the fractionating column. A tiny droplet condenses on the surface and then it re-vaporizes and travels to a new spot on the surface. Each time, the distillate becomes richer in the component with the higher vapor pressure (lower boiling point). After many such occurrences, the mixture is greatly enriched in the lower boiling component. This concept is illustrated in Figure 16-7.
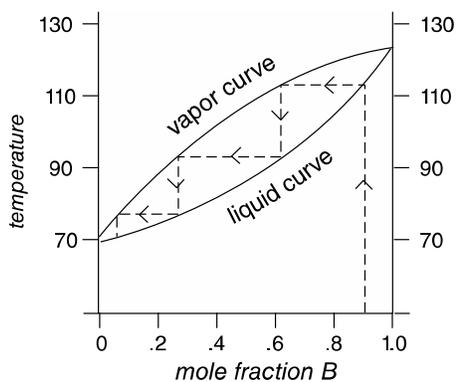

Figure 16-7: In a fractional distillation, several vapor-liquid equilibria are present.

Consider a mixture of mole fraction 0.9 B. After one simple distillation, the "drop" would be about 0.6 B; after two, it would be 0.3 B, and after three it would be less than 0.1 mole fraction B. This means that

the distillate will be mostly A, the lower boiling component, after three simple distillations. Since these simple distillations are taking place in the fractionating column, the distillate is greatly enriched in component A after only one fractional distillation.

As shown graphically in Figure 7.6, the efficiency of a fractional distillation depends on a number of simple distillations that can take place on its surfaces. The efficiency is given by its number of theoretical plates: a theoretical number that signifies the number of vaporization-condensation cycles that occur as the liquid mixture travels through it. The illustration in Figure 16-7 would correspond to three theoretical plates in a fractionating column. The more theoretical plates a column has, the more efficient it is. For the same type of column, a longer column will always have more theoretical plates, simply because it has a larger interior surface area.

Examples of fractionating columns are shown in Figure 16-8. They can either be a specialized piece of glassware, such as a Vigreux column, or a water or air condenser packed with glass beads, glass helices, or stainless steel sponge. In the teaching labs, we use a condenser packed with glass beads.

Jacketed condenser - pack with glass beads, glass helices or stainless steel rings

Air condenser - pack same as jacketed condenser

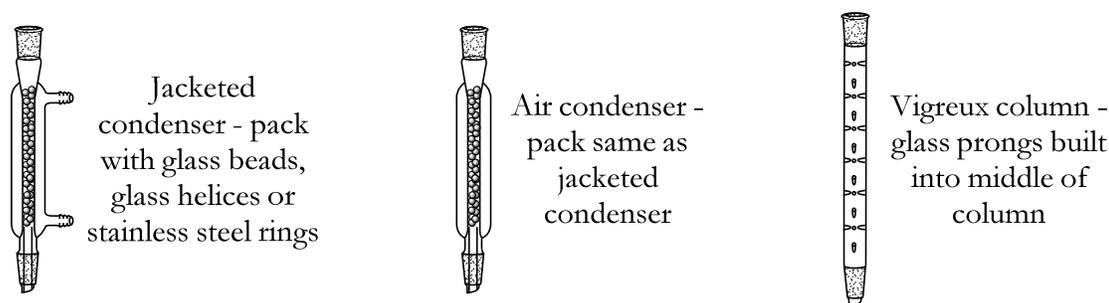Vigreux column - glass prongs built into middle of column

Figure 16-8: Different types of fractionating columns.

The procedure for performing a fractional distillation is very similar to the procedure for simple distillation. The only difference in apparatus is the use of a fractionating column (Figure 16-6). Fractionating columns are subject to flooding, which means that a pool of condensate collects in the column. Flooding occurs when fractionating columns are too tightly packed, or when they are subject to rapid heating or to rapid cooling. If your column floods, remove the heat source until all of the liquid pool has returned to the pot and begin again with a lower heat input. You may also need to insulate the column by wrapping it with aluminum foil. If the column still floods, try a different column that is less tightly packed.

One more difference between simple and fractional distillation is that you cannot fill the round-bottom flask from above. This will lead to less-pure product, since it would involve pouring liquid down through the fractionating column.

C. Steam Distillation

Steam distillation is the distillation of a mixture of water – steam – and an immiscible organic compound. The mixture will boil below 100°C because an immiscible mixture does not behave like an ideal solution (a mixture of miscible liquids). In a mixture of immiscible liquids, the total vapor pressure is the sum of the vapor pressures of the pure individual components. Thus for a steam distillation:

$$P_{total} = P°_A + P°_B$$

and the total vapor pressure equals atmospheric pressure and the mixture boils at a lower temperature than the boiling point of either of the components alone. Since one of the components is always water, the mixture will always boil below 100°C.

Compare the above relationship with that of a solution. In a solution (miscible liquids), the total vapor pressure is the sum of the partial vapor pressures of the components.

$$P_{total} = X_A P°_A + X_B P°_B$$

Note that in a steam distillation, the mole fraction of the components in the mixture is not important. The number of moles of each component in the vapor and thus in the distillate is proportional to the vapor pressure of the pure components.

$$\frac{\text{moles of A}}{\text{moles of B}} = \frac{P°_A}{P°_B}$$

Since the water-organic compound mixtures boil below 100°C, steam distillations are useful for distilling high boiling compounds and for compounds that decompose at high temperatures.

When carrying out a steam distillation, water must be replenished during the course of the distillation. This is typically accomplished by the slow addition of water from an addition funnel (Figure 16-9). Other options for addition of steam include the use of a multi-neck round-bottom flask so that water can be added directly to the distillation pot.
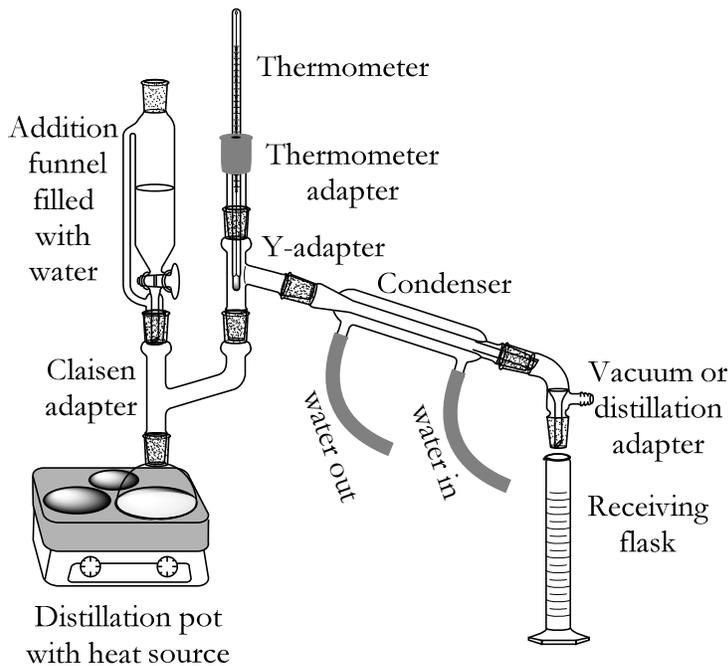

Figure 16-9: Setup for steam distillation.

D. Vacuum Distillation

Vacuum distillation is distillation at a reduced pressure. Since the boiling point of a compound is lower at a lower external pressure, the compound will not have to be heated to as high a temperature in order for it to boil. Vacuum distillation is used to distill compounds that have a high boiling point or any compound that might undergo decomposition on heating at atmospheric pressure. The vacuum is provided either by a water aspirator or by a mechanical pump.

bp, reduced pressure
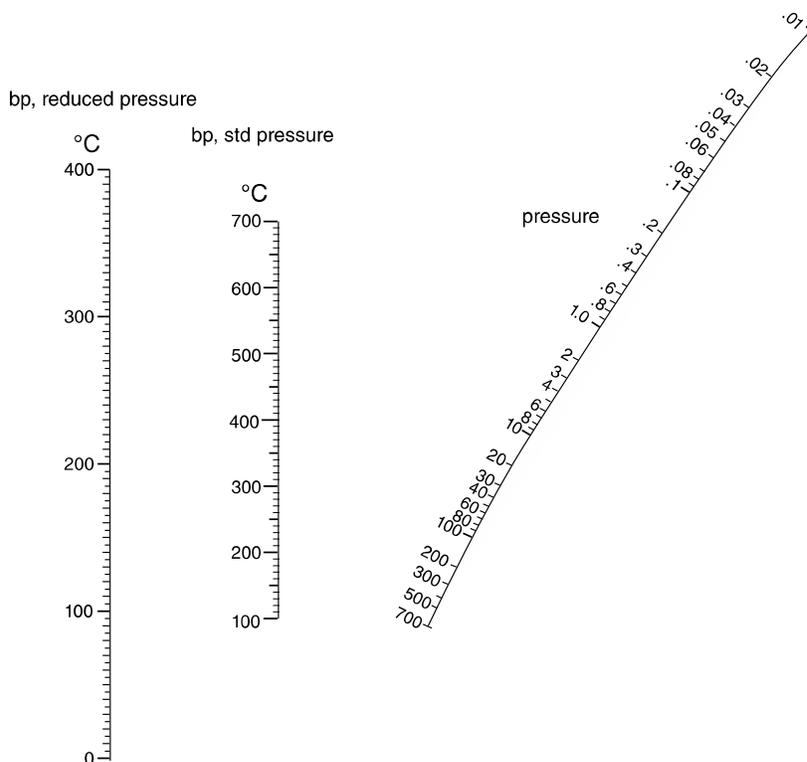
bp, std pressure

pressure

Figure 16-10: A nomogram, which makes it possible to calculate boiling points at reduced pressures.

The pressure-temperature nomogram, or nomograph (Figure 16-10), allows you to use a straightedge to determine the boiling point of a compound at a reduced pressure.

For example, say you are going to distill methyl anthranilate by vacuum distillation. You know that the boiling point of this compound at sea level is 256°C, and you know that the pressure in your vacuum distillation apparatus is 24 torr. Place a straightedge on the nomogram so that it rests on the oblique curved pressure line at "24" and on the vertical normal boiling point line at "256". Draw a line connecting these two points; this line crosses the vertical reduced pressure boiling point line at "142," therefore the boiling point of methyl anthranilate at 24 torr will be approximately 142°C.

The nomogram can be used for estimating reduced-pressure boiling points from normal boiling points as well as estimating normal boiling points from reduced-pressure boiling points.

Vacuum distillations require a closed system with no leaks to the atmosphere. The glassware arrangement is basically the same as a simple distillation with a round-bottom flask securely clamped to the vacuum adapter (see Figure 16-11). Check all glassware for star cracks before assembling, since under the reduced pressure it can implode violently. Make sure all of the connections are tight. To prevent bumping during distillation, place a stir bar in the distilling flask and a stir motor under the heating source and stir during the distillation, or use a microporous carbon boiling chip (regular boiling chips do not work well under reduced pressure).

Connect the vacuum source to the vacuum adaptor using a thick-walled rubber hose. If a vacuum gauge is available, you can connect that to the vacuum source as well. Apply the vacuum before you begin heating the distillation pot. As the vacuum is applied, the liquid in the pot may begin bubbling and frothing; this should subside soon. Once full vacuum is achieved, begin heating the flask until distillate begins condensing in the receiving flask. Record the temperature and pressure at which the distillate is collected.

To discontinue a vacuum distillation, **always** allow the apparatus to return to room temperature before breaking vacuum. Compounds remaining in the pot may be heated to the point where they burst into flame if they are exposed to air. Once the apparatus is cooled, break the vacuum by pulling the vacuum tube from the apparatus before turning off the vacuum source.
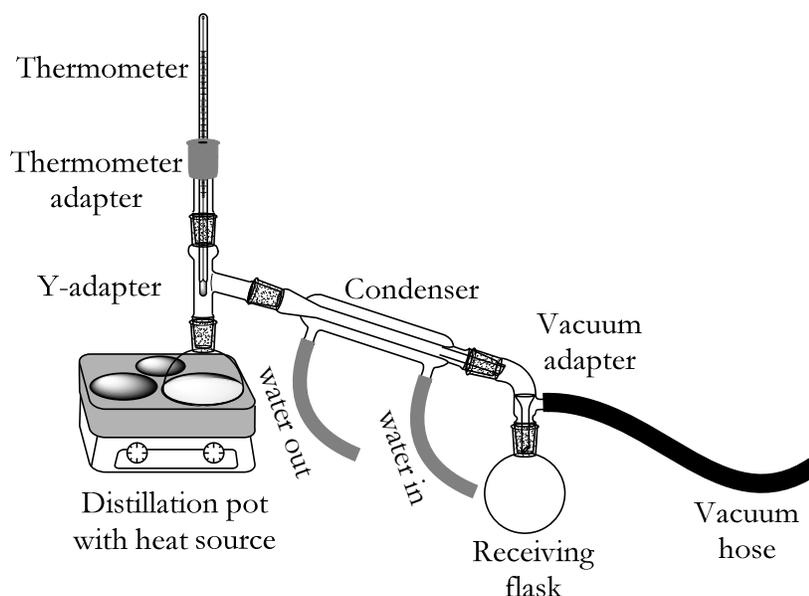

Figure 16-11: Setup for vacuum distillation.

16.3 Azeotropes

Certain mixtures of compounds do not behave as ideal solutions – they do not follow Raoult's law. Sometimes the attractions or repulsions between the molecules of the components of the mixture result in a phenomenon known as azeotropic behavior. An azeotrope is a mixture with a fixed composition that boils at a point below or above the boiling point of either component of the mixture. It behaves as if it were pure, boiling at a constant temperature, but analysis of the distillate reveals that it is a binary mixture. It is difficult to predict whether a mixture will form an azeotrope.

If the azeotrope has a boiling point below that of either component, it is called a minimum boiling point azeotrope. This is the more common type of azeotrope in organic chemistry and results from a slight incompatibility or repulsion between the liquids being mixed. A hypothetical boiling point diagram of this type of azeotrope is shown in Figure 16-12 for two compounds A and B. As the azeotropic mixture is heated, it will begin to boil at the boiling point of the azeotrope. The composition of the boiling mixture in this example is 0.7 mole fraction B. The mixture will continue to boil at this temperature until all of one component in the azeotrope is used up, and then the temperature will rise to the boiling point of the other component.
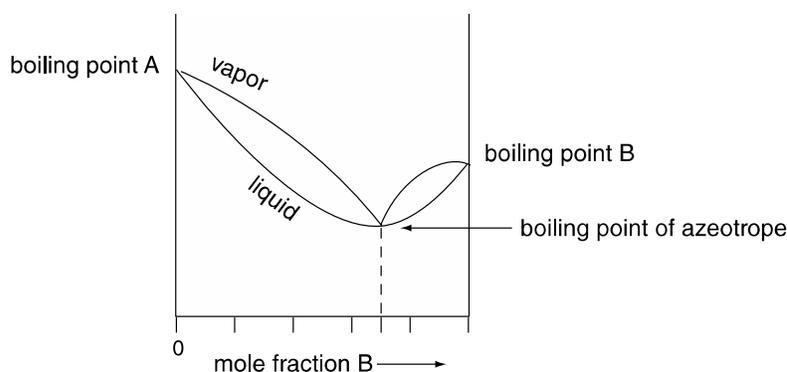
Figure 16-12: Boiling point diagram for azeotropes.

A common minimum boiling point azeotrope is ethanol-water. This azeotrope has a composition of 96% ethanol and 4% water and boils at 78.1°C. This boiling point is just slightly lower than that of pure ethanol, 78.3°C. It means that it is impossible to obtain pure ethanol from the distillation of any ethanol-water mixture that contains more than 4% water, even with the best fractionating column. This is the reason the ethanol in the labs reads "95% ethanol." To obtain it in greater purity, benzene is added to the distillation mixture, producing a ternary benzene-water-ethanol azeotrope that boils at 65°C, and then removing the excess benzene. The resultant product is called "absolute" ethanol, and it is almost 100% pure. (Note that benzene is toxic and the traces that remain render the alcohol unfit for human consumption.)

Although minimum boiling azeotropes are the most common type of azeotrope in organic chemistry, maximum boiling point azeotropes are sometimes observed. They occur when there is a slight attraction between the component molecules. Instead of boiling at a minimum temperature, the constant boiling mixture boils at a value higher than either of the pure components.

A few frequently-encountered azeotropes are listed in Table 16-1.

Table 16-1: Some common azeotropes.

Compounds	Composition (%)	BP
Water & ethanol	4/96	78.1
Water & ethyl acetate	8.5/91.5	70.4
Water & pentane	1.4/98.6	34.6
Water & aniline	80.8/19.2	98.6
Ethanol & cyclohexane	29.2/70.8	64.8
Isopropyl alcohol & hexane	23/77	62.7

16.4 Study Problems

1. The literature reports a boiling point for cyclohexanol of 161°C. In Boulder, Colorado, the atmospheric pressure is usually about 625 mm Hg. In Boulder, what is the observed boiling point of cyclohexanol?

2. You have been given an unknown compound by your TA and told only that it is an ester. You determine the boiling point by simple distillation to be 133°C. The atmospheric pressure is 625 mm. What is the corrected boiling point of the compound?

3. Which of the following compound pairs could be separated by simple distillation?

    a. acetone and aniline

    b. butyl acetate and butanol

    c. cyclohexane and cyclohexanol

    d. hexanes and toluene

4. Refer to Figure 16-4. If the mole fraction of B in the liquid is 0.8, what is the composition of B in the vapor above the liquid? What will be the reading on the thermometer at this point?

5. You prepare a fractionating column by filling the condenser in your lab drawer with glass beads. The glassware in the teaching labs is not entirely uniform from one student drawer to the next, and you notice that when your neighbor packs his condenser with glass beads, he is able to fit a lot fewer beads into the condenser than you are. Whose column will be more efficient as a fractionating column, and why?

6. Why is a fractionating column packed with glass beads more efficient than a Vigreux column of the same length and same diameter?

7. While distilling 50 mL of aniline that you prepared in the lab by a procedure involving an aqueous reaction, you record a boiling point (in Colorado) of 92°C. The temperature remains at this value throughout most of the distillation. However, the distillate drips into the receiving flask as cloudy drops, and separates in the flask as two layers. Explain.

8. Eugenol (clove oil) has a reported boiling point of 255°C at 760 mm Hg. What will be the boiling point under:

    a. Water aspirator vacuum, about 25 torr?

    b. mechanical pump vacuum, about 0.1 torr?