1. Resolution of Mixtures 1: Filtration and Distillation

Objective

The separation of mixtures into their constituent components defines an entire subfield of chemistry referred to as **separation science**. In this experiment, techniques for the resolution of mixtures of solids and liquids will be examined.

Introduction

Mixtures occur very commonly in chemistry. When a new chemical substance is synthesized, for example, oftentimes the new substance first must be *separated* from a mixture of various side-products, catalysts, and any excess starting reagents still present. When a substance must be isolated from a natural biological source, the substance of interest is generally found in a very complex mixture with many other substances, all of which must be removed. Chemists have developed a series of standard methods for resolution and separation of mixtures, some of which will be investigated in this experiment. Methods of separation based on the processes of chromatography are found in experiment "Resolution of Mixtures 2: Chromatography."

Mixtures of solids often may be separated on the basis of differing *solubilities* of the components. If one of the components of the mixture is very soluble in water, for example, while the other components are insoluble, the water-soluble component may be removed from the mixture by simple *filtration* through ordinary filter paper. A more general case occurs when *all* the components of a mixture are soluble, to different extents, in water or some other solvent. The solubility of substances in many cases is greatly influenced by *temperature*. By controlling the temperature at which solution occurs, or at which the filtration is performed, it may be possible to separate the components. For water-soluble solutes, commonly a sample is added to a small amount of water and heated to boiling. The hot sample is then filtered to remove completely insoluble substances. The sample is then cooled, either to room temperature or below, which causes crystallization of those substances whose solubilities are very temperature-dependent. These crystals can then be isolated by a second filtration, and the filtrate remaining can be concentrated to reclaim those substances whose solubilities are *not* so temperature-dependent.

Mixtures of liquids are most commonly separated by *distillation*. In general, **distillation** involves heating a liquid to its boiling point, then collecting, cooling, and condensing the vapor produced into a separate container. For example, salt water may be desalinated by boiling off and condensing the water. For a mixture of liquids, however, in which several of the components of the mixture are likely to be volatile, a separation is not so easy to effect. If the components of the mixture differ reasonably in their boiling points, it may be possible to separate the mixture simply by monitoring the temperature of the

vapor produced as the mixture is heated. The components of a mixture will each boil in turn as the temperature is gradually raised, with a sharp *rise* in the temperature of the vapor being distilled indicating when a new component of the mixture has begun to boil. By changing the receiving flask at this point, a separation will be accomplished. For liquids whose boiling points only differ by a few degrees, the mixture can be passed through a **fractionating column** as it is being heated. Fractionating columns generally are packed with glass beads or short lengths of glass tubing that provide a large amount of surface area to the liquid being boiled. In effect, a fractionating column permits a mixture to be redistilled repeatedly while in the column, allowing for better separation of the components of the mixture.

SAFETY PRECAUTIONS

- **Wear safety glasses at all times while in the laboratory.**
- **The solid mixture contains benzoic acid, which may be irritating to the skin and respiratory tract. Never ingest any chemical in the lab.**
- **When moving hot containers, use metal tongs or a towel to avoid burns. Beware of burns from steam while solutions are being heated.**
- **The liquids used for fractional distillation may be flammable. Keep the liquids away from open flames. Perform the distillation in the exhaust hood to help remove fumes. The liquids may be toxic if absorbed through the skin or inhaled.**
- **Portions of the distillation apparatus may be very hot as the distillation takes place.**
- ***Never heat a distillation flask to complete dryness.*** **The distillation flask may break. Distillation to dryness also poses an explosion hazard for certain unstable organic substances, or for substances which may be contaminated with organic peroxides.**
- **Dispose of all solids and liquids as directed by the instructor.**
- **Silver nitrate, $AgNO_3$, stains the skin. All nitrates are strong oxidizers, toxic, and may be carcinogenic.**

Apparatus/Reagents Required

Impure benzoic acid sample (benzoic acid which has been colored with charcoal), 1% sodium chloride solution, 0.1 *M* silver nitrate, unknown mixture of two volatile liquids for fractional distillation

Procedure

Record all observations and data directly in your notebook in ink.

A. Resolution of a Solid Mixture

Obtain a sample of impure benzoic acid for recrystallization. Benzoic acid is fairly soluble in hot water, but has a much lower solubility in cold water. The benzoic acid has been contaminated with charcoal and sand, which are not soluble under either temperature condition. Transfer the benzoic acid sample to a clean 150-mL beaker.

Set up a short-stem gravity funnel in a small metal ring clamped to a ring-stand. Fit the filter funnel with a piece of filter paper folded in quarters to make a cone. See Figure 1-1.

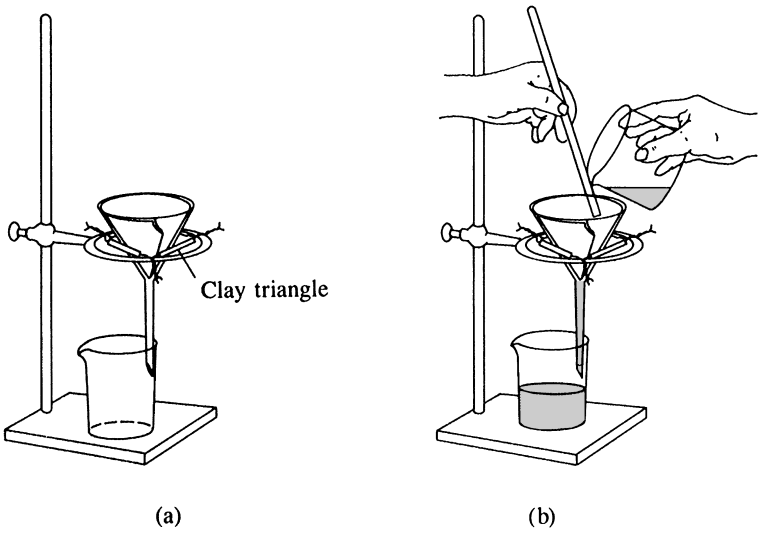

FIGURE 1-1
Filtration of a hot solution. Use a stirring rod as a guide for running the solution into the funnel. Do not fill the funnel more than half-full at a time, to prevent solution being lost over the rim of the paper cone.

Moisten the filter paper slightly so that it will remain in the funnel. Place a clean 250-mL beaker beneath the stem of the funnel.

Set up a 250-mL beaker about half filled with distilled water on a wire gauze over a metal ring. Heat the water to boiling.

When the water is boiling, pour about two-thirds of the water into the beaker containing the benzoic acid sample. Use a towel to protect your hands from the heat.

Pour the remainder of the boiling water through the gravity funnel to heat it. If the funnel is not preheated, the benzoic acid may crystallize in the stem of the funnel rather than passing through it. Discard the water that is used to heat the funnel.

Transfer the beaker containing the benzoic acid mixture to the burner and re-heat it gently until the mixture just begins to boil again. Stir the mixture to make sure that the benzoic acid dissolves to the greatest extent possible.

Using a towel to protect your hands, pour the benzoic acid mixture through the preheated funnel. Catch the filtrate in a clean beaker.

Allow the benzoic acid filtrate to cool to room temperature.

When the benzoic acid solution has cooled to room temperature, filter the crystals to remove water. Wash the crystals with two 10-mL portions of cold water.

Transfer the liquid filtrate from which the crystals have been removed to an ice bath to see if additional crystals will form at the lower temperature. Examine, but do not isolate, this second crop of crystals.

Transfer the filter paper containing the benzoic acid crystals to a watch glass, and dry the crystals under a heat lamp or over a 400-mL beaker of boiling water. You can monitor the drying of the crystals by watching for the filter paper to dry out as it is heated. If a heat lamp is used, do not let the paper char or the crystals melt.

When the benzoic acid has been dried, determine the melting point of the recrystallized material using the method discussed in experiment "The Determination of Melting Point." Compare the melting point of your benzoic acid with that indicated in the handbook. If the melting point you obtain is significantly lower than that reported in the handbook, dry the crystals for an additional period under the heat lamp or over the hot water bath.

B. Simple Distillation

Simple distillation can be used when the components of a mixture have *very different* boiling points. In this experiment, a partial distillation of a solution of sodium chloride in water will be performed (the distillation is not carried to completion to save time). This is an extreme example, since the boiling points of water and sodium chloride differ by over 1000°C, but the technique will be clearly demonstrated by the experiment.

Your instructor has set up a simple distillation apparatus for you. (See Figure 1-2.) He or she will explain the various portions of the apparatus and will demonstrate the correct procedure for using the apparatus. The source of heat used for the distillation may be a simple burner flame, or an electrical heating device (heating mantle) may be provided. Generally electrical heating elements are preferred for distillations, because often the substances being distilled are flammable.

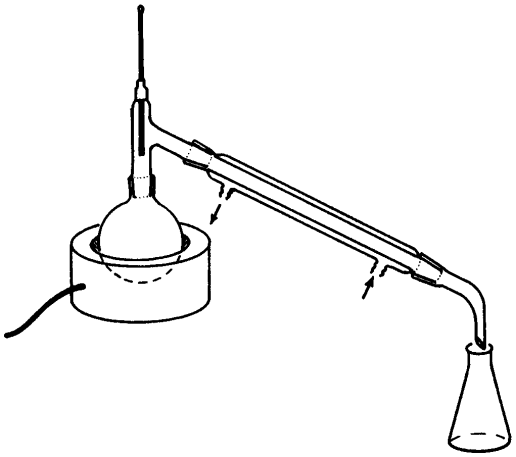

FIGURE 1-2
Simple distillation apparatus. Cold water entering the lower inlet of the condenser causes the vapor being distilled to liquefy.

Obtain about 50 mL of 1% sodium chloride solution. Place 1 mL of this solution in a small test tube, and transfer the remainder of the solution to the distilling flask.

Place a clean dry beaker under the mouth of the condenser of the distillation apparatus to collect the water as it distills from the salt solution.

Begin heating the sodium chloride solution as directed by the instructor, and continue distillation until approximately 20 mL of water has been collected. Transfer approximately 1 mL of the distilled water to a clean small test tube.

To demonstrate that the distilled water is now free of sodium chloride, test the sample of original 1% sodium chloride solution that was reserved before the distillation, as well as the 1-mL sample of water that has been distilled, with a few drops of 0.1 *M* silver nitrate solution (*Caution!*). Silver ion forms a *precipitate* of insoluble AgCl when added to a chloride ion solution. No precipitate should form in the water that has been distilled.

C. Fractional Distillation

Fractional distillation may be used to separate mixtures of volatile substances that differ by at least several degrees in their boiling points. The vapor of the liquid being boiled passes into a *fractionating column,* which provides a great deal of surface area and the equivalent of many separate simple distillations.

Your instructor has set up a fractional distillation apparatus in one of the exhaust hoods. (See Figure 1-3.) Compare the fractional distillation apparatus with the simple distillation apparatus used in Part B, and note the differences. Your instructor will explain the operation of the fractional distillation apparatus. The apparatus is set up in the hood, since the mixture you will distill is very volatile and may be flammable.

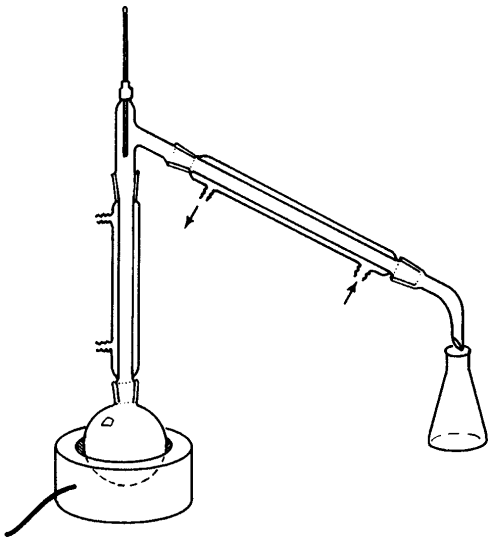

FIGURE 1-3
Fractional distillation. The tall vertical fractionating column is packed with bits of glass that provide a large surface area.

Obtain an unknown mixture for fractional distillation and record its identification number. Use a graduated cylinder to transfer 40 mL of the unknown mixture to the distillation flask. Record the exact volume of the mixture used. During the distillation, carefully watch the thermometer that is part of the apparatus. The temperature is used to monitor the distillation, since the temperature will *increase very suddenly* as one component finishes distilling, and another component begins to distill.

Place a clean dry flask under the mouth of the condenser to collect the first component of the mixture as it distills. Have ready a second clean dry flask for collection of the second component. Have ready corks or rubber stoppers that fit snugly in the two collection flasks.

Have your instructor approve the apparatus, and then begin heating the distillation flask with very low heat until vapor begins to rise into the fractionating column.

Allow the vapor to rise to the level of the thermometer bulb, and adjust the heat so that the thermometer will remain bathed in droplets of liquid as the mixture distills. Record the temperature indicated by the thermometer as the first component of the mixture begins to distill. Collect the distillate coming from the condenser.

Continue heating the distillation flask, using the smallest amount of heat that will maintain distillation. Monitor the temperature constantly. At the point at which the first component of the mixture has finished distilling, the temperature will rise *suddenly* and abruptly by several degrees. At this point, remove the flask used to collect the first component of the mixture, and replace it with the second flask. Stopper the flask containing the first component to prevent its evaporation.

Record the temperature indicated by the thermometer as the second component of the mixture begins to distill. Continue the distillation *until approximately 5 mL of liquid remains in the distillation flask.* Remove the source of heat, but do not remove the collection flask until distillation stops.

Do not heat the distillation flask to complete dryness, or it may break from the heat. When distillation is complete, stopper the flask containing the second component of the mixture.

With a graduated cylinder, determine the respective volumes of each of the two components of the mixture. Calculate the *approximate composition* of the original mixture, in terms of the percentages of low-boiling and high-boiling components. This percentage is only approximate, since some of the vapor being distilled may have been lost, and not all of the high-boiling component was isolated.

Report the approximate composition of your mixture to the instructor, along with the boiling temperatures of the two components.

Turn in the two flasks of distillate to the instructor for proper disposal.

Resolution of Mixtures 1: Filtration and Distillation

Date:	.....	Student name:	.....
Course:	.....	Team members:	.....
Section:	.....		.....
Instructor:	.....		.....

Prelaboratory Questions

1. This experiment examines the techniques of *recrystallization* of solids, and also the techniques of simple and fractional *distillation* of liquids, as examples of methods by which mixtures are resolved into their components. Use your textbook to find three additional methods by which mixtures may be resolved, and describe the techniques briefly, including the sorts of mixtures to which the techniques are applied.

2. Why is a fractionating column packed with small glass beads or short pieces of glass tubing? How does this help improve a distillation?

Resolution of Mixtures 1: Filtration and Distillation

Date: ..... Student name: .....

Course: ..... Team members: .....

Section: .....

Instructor: .....

Results/Observations

A. Resolution of a Solid Mixture

Observation of dissolving sample in hot water

Observation of material remaining on filter paper

Observation of filtrate as it cools

Appearance of crystals collected

Appearance of second crop of crystals

Melting point of dry benzoic acid .....

Literature value for melting point .....

Error in melting point determination .....

B. Simple Distillation

Observation of distillation

Silver nitrate test:

On original sample .....

On distilled sample .....

C. Fractional Distillation

ID number of mixture for fractional distillation .....

Observation of fractional distillation

Boiling point of first (low boiling) component .....

Volume of first component collected .....

Boiling point of second (high boiling) component .....

Approximate volume of second component .....

Approximate percentage composition of mixture .....

Questions

1. The solid in Part A consisted primarily of one component (benzoic acid), with a small amount of an insoluble contaminant added. How might a solid mixture containing *two* major components be separated?

2. How do simple and fractional distillation *differ*? Under what circumstances is one method likely to be used in preference to the other method?

3. Fractional distillation can be used to separate liquid mixtures whose components have different boiling points in many, but not all, instances. The ordinary ethyl alcohol used in laboratories is actually only 95% ethyl alcohol, with the remainder of the mixture being water. Even very careful distillation of 95% ethyl alcohol does not permit removal of the water, even though the boiling points of water and pure ethyl alcohol differ by more than 20°C. Use a chemical encyclopedia to find the definition of an *azeotrope*, and why azeotropes such as 95% ethyl alcohol cannot be separated by distillation. Record your findings here.

4. In spite of such a major portion of the earth's surface being covered by water, surprisingly little use has been made of distillation of seawater as a source of drinking water in arid areas. Why do you suppose this is so?