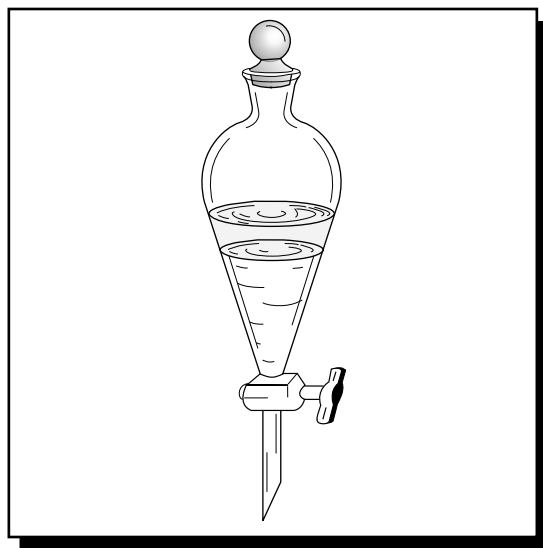


A SourceBook Module

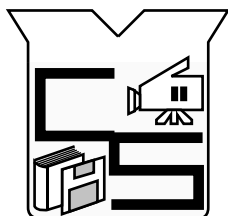
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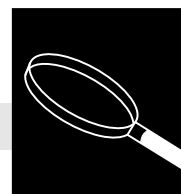
SEPARATIONS



ChemSource

*Instructional Resources for Preservice and
Inservice Chemistry Teachers*

Topic Overview



CONTENT IN A NUTSHELL

Very few compounds occur pure in nature. They must first be mined or extracted and then *purified*. If a particular substance cannot be mined or extracted, then it must be *synthesized*. In any case, it is important to be able to *separate* desired compounds from others that are usually present. Although it does not sound exciting or seem glamorous, it is nonetheless, important in the modern practice of chemistry to be able to obtain pure compounds from the mixtures in which they occur. Four methods of separating the components of mixtures will be developed through laboratory activities. These are *filtration, fractional crystallization, fractional distillation, and chromatography*.

Separations involve the application of chemical principles, properties, and techniques to separate specific elements and compounds from mixtures. Such separations take advantage of differences in solubility, volatility, adsorbability, extractability, stereochemistry of isomers, and the properties of ions, elements, and molecules.

The mixtures to be separated may be either *heterogeneous* or *homogeneous*; in either case, suitable techniques are available to effect a satisfactory separation. Until fairly recently, many substances were scarce because suitable separation techniques had not been developed. For example, until the development of suitable chromatographic separation methods, the similar chemical properties of rare-earth (lanthanide) oxides did not allow a suitable separation. Now these oxides are available in quantity for a variety of uses.

PLACE IN THE CURRICULUM

This module probably does not stand alone in most chemistry courses; it may be partitioned into pieces to fit a particular need. For example, when considering physical properties, it might be well to include separations because the ability to separate depends upon differences in physical properties. Parts of the module may also fit into sections involving qualitative analysis, forensic chemistry, solutions, gases, mole, chemistry in medicine, and so forth. The possibilities are almost limitless.

CENTRAL CONCEPTS

1. Separation of two or more components of a mixture depends upon differences in physical properties. To effect such separations, a knowledge of the physical properties of the substances in a mixture is essential.
2. Filtration may be used for separation when one of two substances is insoluble in a given solvent by dissolving the soluble substance and filtering. The soluble substance passes through the filter but the insoluble one does not, *e.g.*, a mixture of sugar and powdered sulfur may be mixed with water, dissolving the sugar, which then can be passed through a filter with the sulfur remaining behind.
3. Fractional crystallization may be used for the separation of two substances with fairly large differences in solubility at a given temperature, *e.g.*, NaClO_3 (sodium chlorate) and NaCl (sodium chloride) at 0°C have quite different solubilities allowing most of the NaClO_3 to crystallize with the NaCl remaining in solution.
4. Distillation may be used to separate a mixture of volatile and nonvolatile substances, *e.g.*, a salt water solution.

5. Fractional distillation utilizes the difference in boiling points of liquids, although some simple binary (two-component) mixtures form constant boiling azeotropes. For example, a mixture of 2-propanol and water may be separated into alcohol- and water-rich fractions.
6. Paper and column chromatography separate mixtures through differences in solubility in the developing solvent and differences in adsorptivity between the substances in the mixture and the developing substrate. For example, a mixture of food dyes may be separated using paper chromatography, or a mixture of chlorophylls in a green leaf may be separated using column chromatography.

1. Solutions are homogeneous mixtures with varying degrees of interaction between solute and solvent depending upon their nature, *i.e.*, polar or nonpolar.
2. Solubilities of substances is quite variable and depends, among other factors, upon the degree of interaction between solvent and solute. These differences provide a means of separation of substances.
3. Adsorption is a surface phenomenon that is useful for effecting separations.
4. Volatile substances vaporize easily while nonvolatile ones do not.
5. Melting and boiling points give an indication of the degree of interaction between the molecules and/or ions of substances.

Manipulative laboratory skills involving the use of common glassware and hardware are needed to perform the activities.

After completing their study of separations, students should be able to:

1. describe the separation techniques used to isolate the components of mixtures and how to measure the amounts of the components. The techniques include but are not limited to: filtration, fractional crystallization, simple distillation, fractional distillation, and chromatography.
2. perform simple separations in the laboratory including filtration, distillation, crystallization and fractional crystallization, and chromatographic techniques (paper, thin layer, column, *etc.*).
3. describe some problem-solving applications of separations.
4. explain the importance of separation of components of mixtures to the maintenance of our quality of life and leisure time, particularly the importance of the separation techniques in industry and production of the commodities of commerce.
5. describe some of the applications of separation techniques, *e.g.*, quality control, consumer protection, pollution monitoring.
6. describe some uses of separations in their communities, *e.g.*, water quality testing, sewage treatment, medical applications, *etc.*
7. list some of the vocational and career opportunities available that are based on separation techniques.

RELATED CONCEPTS

RELATED SKILLS

PERFORMANCE OBJECTIVES

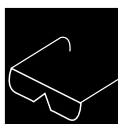
Concept/Skills Development



LABORATORY

ACTIVITY: Introduction

STUDENT VERSION



Activity 1: Separating Mixtures

To determine the chemical and physical properties of elements and compounds, chemists need to be sure that the substances are pure. However, most substances in nature are mixtures and must be separated into their components. To effect such separations, it is possible to use differences in properties by employing appropriate techniques. The techniques presented in this activity are fairly representative of those that have proven useful in both the laboratory and in industry for this purpose. Additional techniques may be demonstrated.

Purpose

To illustrate several commonly used techniques for separating the components of a mixture.

Safety

1. Wear protective goggles throughout the laboratory activity.
2. The usual laboratory safety precautions apply to the four procedures presented in this activity. However, particular caution should be exercised with some of the substances used since these may be toxic or flammable.
3. Open flames should not be used when alcohols or other organic solvents are present.
4. Observe the safety rules presented prior to each procedure.

Part I. Gravity Filtration

Purpose

To separate soluble and insoluble substances by dissolving the soluble substance in water, and then separating the insoluble substance from the liquid solution by filtration.

Safety

Treat the mixture to be separated with the usual precaution, *i.e.*, do not taste or ingest the mixture. Dispose of solid in an appropriate container. Do not return excess mixture to its container.

Procedure

1. Obtain an unknown mixture from your teacher. Weigh out 3.00 g unknown to the nearest 0.01 g on a piece of weighing paper. It is not important that exactly three grams is weighed out, but it is important that the weight is known to the nearest ± 0.01 g. Transfer the weighed unknown to a 250-mL beaker. Add 100 mL distilled water and stir.
2. Weigh a filter paper circle to the nearest two decimal places and record the mass. Prepare the filter paper circle by folding it in half, and then again into quarters. Open the filter paper to form a cone with one thickness of paper on one side and three thicknesses on the other. Place the filter paper cone into a filter funnel, place the funnel in a ring on a ringstand, and then moisten the paper with a small volume of distilled

water from a wash bottle. Gently press the paper against the sides of the funnel to make a snug fit by using a glass stirring rod. If correctly selected, the filter paper cone's top edge will be just below the rim of the funnel.

3. Use a 250-mL beaker to collect the **filtrate** (the liquid component of the mixture) by placing the beaker below the funnel tube so that the tube touches the side of the beaker. The funnel tube should extend below the beaker's rim by 2-4 cm. After stirring, pour the mixture from the first beaker down a glass stirring rod into the funnel. Do not allow the liquid level in the funnel to be more than three-fourths of the distance from the bottom of the funnel to the top of the filter paper.
4. When filtration is complete, rinse any remaining solid particles from the first beaker into the funnel by using a wash bottle and distilled water. Pour three 10-mL portions of distilled water over the solid in the filter paper. Remove the beaker with filtrate and replace it with a clean beaker. Then pour three 10-ml portions of acetone over the solid in the filter paper to rinse it free of water. Remove the filter paper and contents. Carefully open the filter paper circle and place it on a paper towel, being careful to not lose any of the solid. Place the paper towel with filter paper and solid under the fume hood to air dry. Pour the rinse acetone from the beaker into the container designated by your teacher for this purpose. Dispose of the filtrate as directed by your teacher.
5. After the filter paper and solid are dry, weigh them to the nearest two decimal places on the laboratory balance. The difference between the mass of solid collected and the original sample allows the composition of the mixture to be determined.
6. Thoroughly wash your hands before leaving the laboratory.

Data Analysis and Concept Development

1. Using the difference in mass between the original unknown sample and the solid obtained after filtration, calculate the percent composition of the original sample as percent by mass of the soluble component of the mixture.
2. Obtain the accepted value from your teacher and calculate percent error as follows:

$$\% \text{ Error} = \frac{\text{Accepted value} - \text{Experimental value}}{\text{Accepted value}} \times 100\%$$

Implications and Applications

1. How efficient is gravity filtration? Explain on the basis of your data.
2. If the filtrate in your activity is not as clear as distilled water, offer a possible explanation.
3. Why should the end of the filter funnel touch the side of the collecting beaker in gravity filtration?

Part II. Fractional Distillation

Purpose

To separate a mixture of two miscible liquids (liquids that mix in all proportions) with different boiling points. Your mixture will consist of water and 2-propanol (isopropyl or rubbing alcohol).



Safety

1. Wear protective goggles throughout the laboratory activity.
2. 2-propanol (rubbing alcohol) is generally safe; however, it is flammable, toxic, and requires care in handling.
3. Use open flames with caution since the alcohol and some alcohol-water mixtures are flammable.
4. Place the alcohol-water mixtures in a designated container.

Procedure

Carefully examine the mixture that is to be separated. Cautiously smell the mixture and note any odor. Dip a piece of filter paper in the mixture to wet one end and try to light it with a match. Carefully measure the density of the mixture using a graduated cylinder and balance. Test the solubility of sugar in a small amount (about 1 mL) of the liquid. Record all observations.

1. Set up a distillation apparatus as shown in Figure 1. Check the following details.

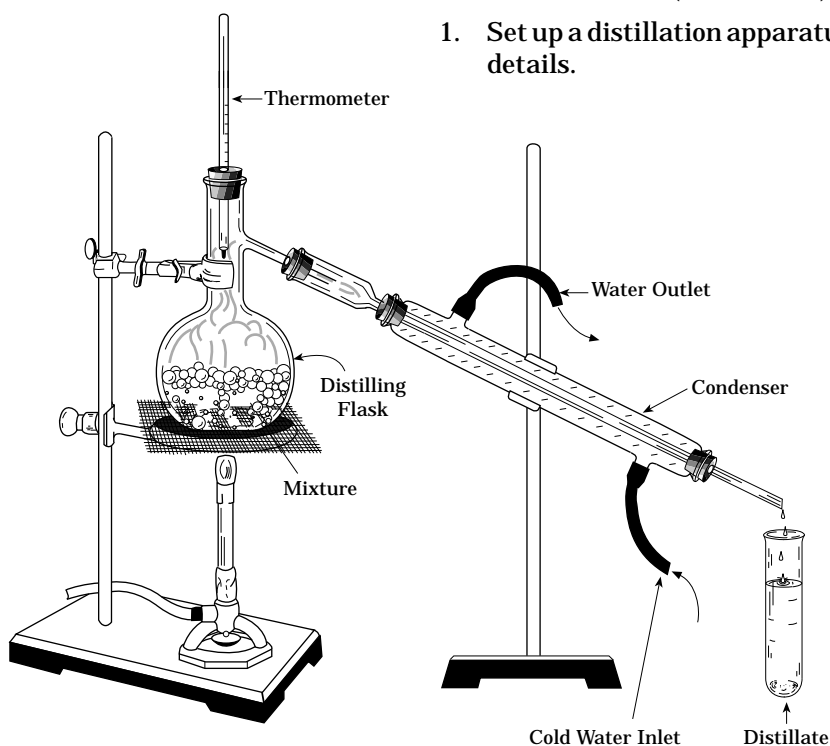


Figure 1. A simple distillation apparatus.

- a. The top of the thermometer bulb should be just below the side arm of the distilling flask, insuring that the entire thermometer bulb will be bathed in the rising vapor as the liquid mixture is heated.
 - b. The side arm should extend beyond the cork in the top end of the condenser, and the condenser tip should extend beyond the cork in the adapter. The distillate can not pick up impurities from the corks when set up this way.
 - c. Both the distilling flask and the condenser must be firmly clamped in place.
 - d. To prevent having a closed system, the lower end of the adapter should not be connected to the receiver by a cork. This arrangement prevents pressure from building up and causing an accident.
 - e. Begin circulating water from the lower end of the condenser prior to heating the distilling flask.
 - f. Add two or three boiling chips (small marble chips or porcelain chips) to the distilling flask to prevent superheating and bumping.
 - g. Have your teacher check the setup before applying heat to the flask.
2. Label and number four large test-tubes (20- x 150-mm) for collecting fractions. Place Tube 1 under the condenser outlet.

3. Remove the thermometer with its cork. By means of a long-stem glass funnel, add about 20-30 mL 2-propanol-water mixture to the distilling flask.
4. Gently heat the flask until the liquid begins to boil. Take temperature readings every half-minute and record these along with the time. Continue heating just enough to keep the liquid boiling while continuing to take temperature readings. Continue boiling the liquid until the temperature remains the same for about three or four minutes.
5. Graphing the data from Step 4 will provide the information needed to decide about the temperature range to collect separate fractions. Do the graphs before continuing. Repeat the procedure (Steps 3-4) but this time use the data collected in Step 4 to collect separate fractions of distillate in Tubes 2, 3, and 4. You should be able to detect the presence of at least three separate fractions, the last one being essentially pure water.
6. Cautiously smell each fraction and record your observations. Test the flammability of each fraction by dipping a small piece of paper in each and trying to ignite the wet paper with a match. Use a balance and graduated cylinder to measure the density of each fraction. Test the solubility of sugar in a small amount (about 1 mL) of each fraction. Record all observations.
7. Thoroughly wash your hands before leaving the laboratory.

Data Analysis and Concept Development

1. Can you tell just by looking that the clear liquid is a mixture?
2. Does the paper dipped in the liquid burn?
3. What is the density of the mixture prior to distillation?
4. What is the density of each fraction?
5. Does sugar dissolve in each fraction?

Implications and Applications

1. What do you conclude about the composition of each fraction?
2. On the basis of the tests performed on the original solution and each fraction, what can you conclude about the composition of each fraction?
3. What do you think would be the result if you redistilled each fraction into three fractions of equal volume?

Part III. Fractional Crystallization

Purpose

To separate a mixture of two solutes dissolved in the same solution by using differences in solubility at a low temperature.

Safety

1. Wear protective goggles throughout the laboratory activity.
2. Sodium chloride and potassium nitrate are generally safe to handle; however, potassium nitrate is an excellent oxidizer and *must not be in contact with organic substances or other oxidizable materials or metals.*



Procedure

1. Examine the solid sample supplied by your teacher. Is it possible to see more than one substance? Record observations.
2. Transfer the solid to a 150-mL beaker. Add about 10 mL distilled water to the mixture, and heat gently while stirring with a glass stirring rod.
3. When the solution boils, add more distilled water in small portions with heating and stirring until the solid just completely dissolves. About 25 mL water should be sufficient. *Do not add too much water or precipitation may not occur.*
4. Add two or three boiling chips, cover with a watch glass, and heat to boiling until about one-half of the water has evaporated. Some spattering may occur but will not affect the the results. While waiting for the water volume to reduce, prepare a funnel and filter paper. Obtain a test-tube large enough to hold the remaining liquid (at least 25- x 150-mm).
5. Just before doing the next step, pour about 15-20 mL hot distilled water through the filter paper and the funnel that has been prepared.
6. Immediately filter the hot solution into a clean, large test-tube. Use beaker tongs or a paper towel folded into a narrow strip encircling the beaker for handling the hot beaker. Scrape any solid remaining in the beaker onto the filter paper.
7. If instructed to do so, allow the filtrate (clear solution) to cool overnight and perform Steps 6-8 in the next laboratory period. Otherwise, place the test-tube containing the filtrate into an ice-water bath in a 400-mL beaker. Allow crystals to precipitate from the solution. When precipitation is complete, remove the test-tube from the water bath, decant the filtrate, scrape the solid out of the test-tube with a stirring rod, and dry the solid on a filter paper circle. Folding the filter paper, placing it between paper towels, and then squeezing expedites removing water from the residue.
8. The original filter paper circle contains mainly sodium chloride while the second filter paper contains mainly potassium nitrate. Compare the appearance of the two solids. Describe any similarity or difference between the two.
9. Place 0.5 g amounts of each solid in two separate large test-tubes and add 2 mL distilled water to each. Shake or stir to dissolve each solid. Then, carefully heat each test-tube to boiling and continue boiling gently until the solutions become saturated, *i.e.*, some solid precipitates out of solution. Qualitatively compare the solubilities of the two solids.
10. Thoroughly wash your hands before leaving the laboratory.

Data Analysis and Concept Development

1. Do the two solids look alike after they have been separated?
2. How do the solubilities of the two solids compare in cold and hot water?
3. Are the two solids two different substances?

Implications and Applications

1. What can you conclude about whether the two solids have been separated?
2. If 100 mL water at 10 °C is saturated in both potassium nitrate and sodium chloride, predict what will happen if the temperature of the solution is raised to 100 °C.

- The solubility of both sodium chloride and potassium nitrate is about 36 g/100 g water at 23 °C, but at 100 °C, the solubilities are about 39 g/100 g water and about 240 g/100 g water, respectively. At 10 °C, the solubilities are about 36 g/100 g water and about 21 g/100 g water, respectively. Why is the solubility difference of these two substances at the temperatures given advantageous in separating the substances?

Part IV. Chromatography

Purpose

To separate the colored substances in an extract from green leaves by column chromatography.

Safety

- Wear protective goggles throughout the laboratory activity.
- The solvents used are highly flammable; open flames must not be used.
- Do not pour solvents into the drain but dispose of them as directed by your teacher.

Procedure

- To prepare a chromatography column, obtain a 25- x 2-cm glass tube, a one hole rubber stopper fitted with a glass tube, a short length of latex rubber tubing to fit the glass tube, a screw (or pinch) clamp, a plug of glass wool, and a short medicine dropper. A slurry made of 50 g of washing soda (sodium carbonate) and TTE (1,1,2-trichlorotrifluoroethane) will be used to pack the column. Assemble the chromatography column as shown in Figure 2.
- Using a ring stand and support clamp, clamp the tube in a vertical position. After the screw (or pinch) clamp is closed, pour a slurry made from 50 g washing soda mixed with just enough TTE to be pourable into the upright tube. Use a thin glass rod to pack the slurry in the tube. It is important that there are no channels between the slurry and the tube. The column of washing soda should be about 20-cm tall. Run excess TTE from the bottom of the tube until the meniscus is just at the surface of the solid. If the column must be stored, it should be stoppered to prevent solvent evaporation.
- Prepare three clean, dry test-tubes for collecting the fractions eluted. Label the test-tubes: xanthophyll and carotenes, Chlorophyll A, and Chlorophyll B.

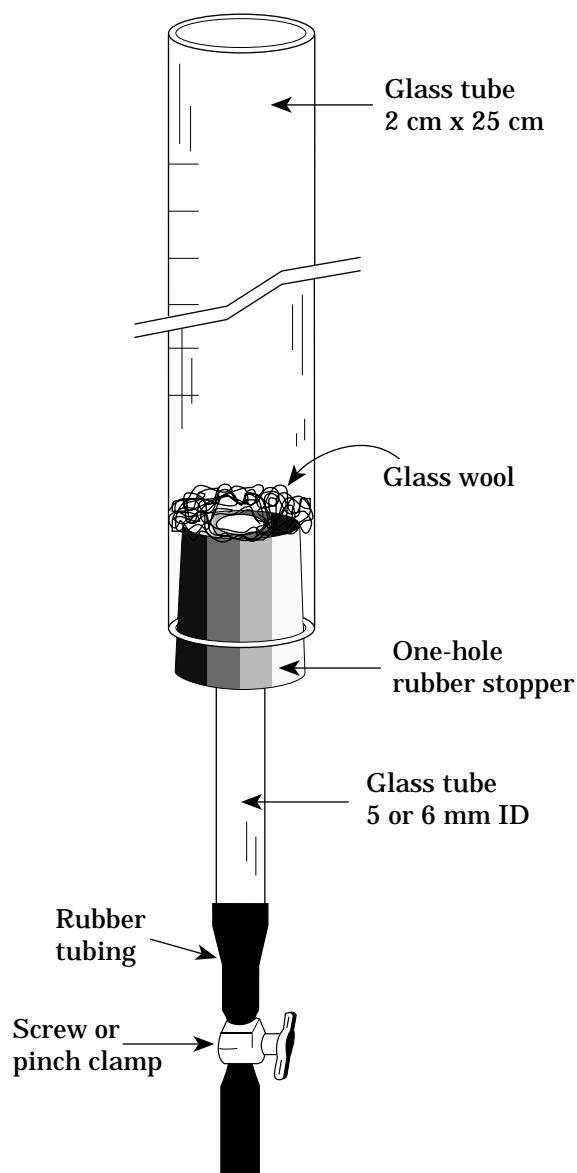


Figure 2. Column chromatography apparatus.



4. Obtain 20 mL green plant extract and *carefully* pour it into the column. Try not to disturb the top of the column. Use a 125-mL conical flask to collect the **eluate** (liquid running from the column), and adjust the clamp for a flow rate of one drop per second.
5. When all of the sample has been adsorbed onto the column, add 10 mL TTE to the top of the column. Two bands of xanthophyll and carotenes should separate from the dark green band at the top and move rapidly down the column. Collect this yellow fraction (about 6 mL) in the test-tube. When the TTE layer is nearly at the top of the column, 10 mL of 10% solution of acetone in TTE is added. A band of chlorophyll A (yellow-green) should separate and move down the column. Collect this fraction (about 7 mL) in a separate, labeled test-tube.
6. After the Chlorophyll A is collected, add 10 mL of 20% acetone in TTE solution to remove Chlorophyll B. Collect this dark green sample in a separate, labeled test-tube.
7. Follow your teacher's directions for cleaning the column.
8. *OPTIONAL:* If directed to do so, confirm the presence of the two chlorophylls by taking an absorption spectrum with a spectrophotometer. Each should show a peak absorption at a characteristic wavelength (see *Instrumentation* module).
9. Thoroughly wash your hands before leaving the laboratory.

Data Analysis and Concept Development

1. How many *different* color bands were observed as the sample of plant pigments traveled through the chromatography column?
2. Was the separation complete or did the bands seem to run together? What might cause the bands to run together?
3. *OPTIONAL:* What was the wavelength of maximum absorption for the two green colored substances when the substances' absorption spectra were taken with a spectrophotometer?

Implications and Applications

1. Why were two different concentrations of acetone in TTE used?
2. Why did the mixture of pigments separate? Consider both solubility and adsorption by the substance in the chromatography tube.
3. It is not unusual to find that some substances that frequently occur together have similar chemical and physical properties, *e.g.*, compounds of the lanthanide and actinide elements. Could chromatography be

useful to separate such substances?

Activity 1: Separating Mixtures

Major Chemical Concept

Instrumental analysis is commonplace in the practice of chemistry. However, it is necessary to have relatively pure samples of substances to analyze. Producing such samples is the role of physical separations since sources of most compounds are rarely pure. After the substance is isolated and purified, instrumentation may be used to study its identity and structure.

Separation procedures presented in this activity are relatively simple and all rely upon a differential physical property. Although procedures presented here are all single step operations, they can be linked to form multi-step operations.

Some mixtures of solids may be separated by dissolving one or more components. For example, a mixture of sand, salt, and iron filings is such a mixture. This mixture is classified as **heterogeneous**. This means it is not uniform but varies in composition from point to point. This fact would be obvious under a microscope. It would be possible to separate the three components of the mixture by using a microscope, tweezers, and a large amount of time and patience. A much more rapid separation is possible, however, by using other methods. A magnet can be used to remove the iron from the mixture leaving the sand and salt. Water can then be added to dissolve the salt, and the resulting solution separated from the sand by **filtration**. The solution passes through the porous filter while the sand does not. Finally, the water may be evaporated to leave the salt residue. This evaporation could be a simple **distillation** that is done by heating the solution in a flask and passing the vaporized water through a condenser. In this fashion the added water may also be collected in fairly pure form.

Distillation can also be used to separate volatile liquids by a process known as **fractional distillation**. In this procedure, the liquid mixture is placed in the flask of a distillation apparatus. As the flask is heated, the lower boiling liquid vaporizes faster than the higher boiling liquid. Thus, although both components are in the vapor phase, the vapor is richer in the lower boiling liquid. The resulting vapor could be condensed and re-evaporated making the resulting vapor still richer in low boiling liquid. If the process is repeated a sufficient number of times, a separation of the two volatile components can be achieved.

Crystallization, or **fractional crystallization**, is a purification method that depends upon the change of solubility of a substance with a temperature change. In practice, when two substances have distinctly different solubilities, this method may be used. For example, mixtures of KNO_3/NaCl and $\text{NaClO}_3/\text{NaCl}$ can be separated this way. The process is based upon the fact that solubilities of KNO_3 and NaClO_3 are much greater than that of NaCl in hot water, but much lower in ice water. If a mixture of either pair is dissolved in hot water, and then the solution is allowed to cool to 0°C , the more soluble NaClO_3 will crystallize out of solution while little if any of the NaCl crystallizes, provided that a sufficiently small amount of NaCl was initially present. To further purify the material, it may be recrystallized. A complete separation of the two substances is not made; however, a large proportion of the more soluble substance is recovered in rather pure form. The remaining solution will contain some of both compounds.

Chromatographic methods may be used to separate liquids and solids. Chromatography constitutes one of the most versatile and powerful separation

LABORATORY ACTIVITY: TEACHER NOTES



techniques used by chemists. The types of chromatography considered here are paper chromatography and column chromatography or liquid-solid chromatography. Other common chromatography types are thin layer chromatography (see *Forensic Chemistry* module) and gas-liquid chromatography. **Paper chromatography** depends upon a piece of hanging paper with one end dipping in a liquid so that the liquid wicks up along the paper. A drop of solution containing a mixture to be separated is placed near the edge of the paper strip dipping in the liquid. When the liquid in contact with the paper wicks upward, it passes over the drop of mixture. The components in the drop that interact least strongly with the paper and most strongly with the liquid travel farther up the paper with the wicking liquid. In this way, components of the mixture are separated.

Level

Procedures I, II, and III may be used with all levels of students. Procedure IV is better used with honor students, and may be demonstrated to students at other levels. The choice of procedure(s) will depend upon your confidence, apparatus and materials available, and students' ability level.

Expected Student Background

Students should have some experience with laboratory apparatus and glassware. These procedures require good manipulative ability. You may need to demonstrate some of the techniques such as filtration and setting up the apparatus. Students should also know about properties such as melting point, solubility, adsorption, *etc.*, since these properties form the basis for the separations in the activity.

Time

Teacher preparation time for each procedure varies between 30 min to 2 hr, depending upon how accessible the equipment is and how much of the equipment you plan to assemble for students. Chemical preparation is at a minimum with either simple solutions to prepare or grinding a mixture of NaCl and KNO₃ together. Students should be able to finish the procedures for simple filtration and fractional crystallization in a single period. The procedures for fractional distillation and column chromatography will each require two laboratory periods (50-55 min).

Safety

Read the *Safety Considerations* in the *Student Version*. Treat all the substances used in these procedures as potentially hazardous. Instruct students about disposal of the solvents. 2-propanol can be safely flushed down the drain with sufficient water. TTE (1,1,2-trichlorotrifluoroethane) should be collected and recycled. All solids may be disposed of in the usual fashion. It is recommended that you collect the potassium nitrate since students might be tempted to take some for use in making gunpowder. Safety goggles must be worn in the laboratory at all times. Instruct students to wash their hands thoroughly prior to leaving the laboratory.

Materials (For 24 students working in pairs)

Part I. Simple Filtration Procedure

Nonconsumables

- Balance, centigram
- 12 Wash bottles
- 24 Beakers, 250-mL
- Distilled water
- 12 Graduated cylinders, 100-mL and 10-mL
- 12 Filtering funnels

12 Glass rods, stirring
12 Spatulas, porcelain

Consumables

12 Filter paper circles
50-50 Mixture NaCl and CaCO₃, 36-48 g
Weighing paper
Paper towels

Part II. Fractional Distillation Procedure

Nonconsumables

12 Distilling flasks, 150-mL
Boiling chips
12 Condensers fitted with rubber tubing
24 Ring stands with support clamps
12 Iron rings
48 Test-tubes, small
12 Stirring rods
12 Wire gauze, ceramic centered
12 Burners
12 Thermometers, -10 °C to 110 °C x 1 °C
12 Adapters to fit condenser (not absolutely essential)
60 Test-tubes, 20- x 150-mm
12 Funnels, long stem
12 Graduated cylinders, 10-mL, 25-mL, and 50-mL
12 Test-tube racks

Consumables

Sugar, 10 g
Boiling chips
Filter paper or other porous paper
2-propanol-distilled-water mixture, 1200 mL (two parts 2-propanol to three parts water)
Matches
Labels
Marking pencils
Graph Paper
Rulers
Pencils

Part III. Fractional Crystallization Procedure

Nonconsumables

24 Beakers, 150-mL and 250-mL
12 Beakers, 400-mL (optional for Step 6 ice-water bath)
Boiling chips
12 Filtering funnels
12 Graduated cylinders, 10-mL
12 Watch glasses (to cover beaker)
12 Stirring rods
36 Test-tubes, 25- x 150-mm

Consumables

24 Filter paper circles to fit funnel
50-50 Mixture of NaCl and KNO₃, 200 g



Distilled water
Boiling chips

Part IV. Chromatography Procedure

Nonconsumables

24 Beakers, 250-mL
12 Conical flasks (Erlenmeyer), 125-mL
12 Glass tubes, 25- x 1- or 2-cm (2 cm diameter is preferred) or 12 Mohr burets, 25- or 50-mL
12 Rubber stoppers, one-hole to fit glass tube
12 Rubber stoppers, solid to fit glass tube
24 Graduated cylinders, 10-mL
12 Short glass tubes to fit one-hole rubber stopper
12 Latex rubber tubes, 4-in length
12 Clamps, screw or pinch to fit rubber tubing
12 Short medicine dropper glass tubes
12 Ringstands with support clamps
36 Test-tubes
12 Stirring rods

Consumables

Sodium carbonate, Na_2CO_3 , 600 g
TTE (1,1,2-Trichlorotrifluoroethane), 2.4 L
Glass wool (small amount for chromatography tube)
Plant extract (see *Advance Preparation*)

Advance Preparation

One activity is presented for use with this topic. It is about separations and provides four procedures including simple filtration, fractional crystallization, fractional distillation, and column chromatography. Any number may be used depending upon your objectives. Another possibility is to divide the class into four groups, have each group perform one of the procedures, and then give a class report. Check to be certain that the safety shower, the fire blanket, the eyewash, and the fire extinguisher are functioning correctly.

Part I

For the simple filtration procedure, prepare a 50-50 mixture of precipitated chalk (CaCO_3) and sodium chloride. (If precipitated chalk is unavailable, grind white chalk to a fine powder in a mortar and pestle.) Allow about 5-6 g per student or group. Grind the sodium chloride in a mortar and pestle until it is powdered. Mix the two substances thoroughly so that the mixture appears to be a single substance.

Part II

Prepare a 2:3 mixture (by volume) of 2-propanol and distilled water. Ordinary rubbing alcohol may be used; however, the rubbing alcohol is a 70% by volume mixture of 2-propanol and water. Thus, approximately equal volumes of the alcohol and water will be about right.

If condensers and distilling flasks are unavailable, a large test-tube fitted with a two-hole rubber stopper having a thermometer and a bent glass tube can be used. The thermometer bulb should be close to the bottom of the stopper and the bent glass tube is fitted with clear plastic tubing that leads into a large test-tube immersed in a beaker of cold water. The water-alcohol mixture is heated in the test-tube in this

alternate setup. Of course, a smaller amount of the mixture to distill will be needed, *i.e.*, only about 40 mL per student or group.

Part III

The unknown mixture is prepared by grinding together in a large mortar and pestle equal masses of reagent grade sodium chloride (do not use common table salt) and potassium nitrate. For safety, make certain that no combustible material is in the mortar prior to grinding. Each group or student should be given about 15 g of the mixture. To simplify this task, place a 15-g sample in a test-tube, and then fill the other test-tubes to the same level. This will save considerable time rather than having each student or group weigh out a sample. Using 100 g of each substance in the mixture is adequate for a class of 24 students working in pairs.

Since the solution must be filtered while hot, the funnel and filter paper need to be preheated with hot distilled water immediately before the solution is filtered. A second burner, ring stand, iron ring, and ceramic centered wire gauze is needed for this purpose, or a large container of hot distilled water can be made available for students to obtain hot water.

Part IV

To prepare the extract, obtain about 10 g of a green plant (frozen spinach works well). Homogenize the green plant with 25 mL acetone in a blender for 1-2 min. (Alternately, the green plant may be ground in a mortar and pestle with some clean sand, and the green pigments extracted with 25-35 mL acetone. The remainder of the procedure is the same after either method of extraction.) The homogenate is then suction filtered (Buchner funnel with fritted glass or filter paper circle to fit), suction flask, tubing, and aspirator. The residue in the funnel is washed with 10 mL acetone. Discard the residue. The filtrate should be a very dark green solution. Extract the filtrate with 30 mL TTE in a large separatory funnel.

After extraction, discard the acetone portion and wash the TTE portion (green color) three times with 10 mL distilled water in the separatory funnel. Discard the water, place the TTE solution in a flask along with 5 g anhydrous Na_2SO_4 to absorb the residual water. Stopper the flask and set aside in a dark place. To save time, the chromatography tubes may be prepared in advance, stoppered, and stored until needed. While in storage, the TTE level in the chromatography tubes should be just above the sodium carbonate in the tube. *NOTE:* In preparing the chromatography column, be sure to make a fine slurry of sodium carbonate and TTE. It should pour easily into the column and be allowed to settle to avoid channeling. Some teachers prefer to use powdered sugar as the stationary phase and hexane as the mobile phase.

Pre-Laboratory Discussion

Select one or two procedures that are within student capabilities. Thoroughly discuss the procedure and demonstrate any new techniques prior to students' laboratory work. Caution students about the flammability of solvents used and the danger of having open flames while solvents are being used. Another possibility is to have different student groups do different procedures and make a group report about the procedure and results. However, do not allow procedures using acetone to be performed next to procedures requiring a burner. Hotplates can be substituted as a source of heat.

Help students with data tables and/or methods of recording observations. None of the procedures require elaborate data tables, so designing them should be within the capability of the student. Point out that measurements must be recorded. It might be sufficient to simply identify the data to be recorded and leave the rest to the student.



Equipment and materials for the laboratory should be easily accessible to students. Demonstrate the assembly of apparatus for *Parts II* and *IV*. For *Part II*, demonstrate the distillation assembly consisting of distilling flask, condenser, and adapter. Include the direction of water flow through the condenser (bottom to top—see Figure 1). Most chemistry texts have solubility graphs showing the solubility of NaCl and KNO₃. You may want to include this in the pre- or post-laboratory discussion for *Part III*.

Teacher-Student Interaction

Circulate to answer student questions and help with equipment setup and technique. Acting as a safety officer is appropriate. Do not hesitate to correct students for unsafe practices. Encourage students to complete assigned procedures and report results regardless of the student's view about correctness of results. If students decide better results can be achieved by repeating the procedure, encourage them to do so if there is time. Providing students with specific uses for separation techniques should be motivating.

Anticipated Student Results

$$\frac{\text{Mass of mixture} - \text{Mass of residue}}{\text{Mass of mixture}} \times 100 = \% \text{ Soluble component}$$

Part I. Gravity Filtration

With reasonable care, students should obtain results within 5-10% of the actual composition of the original mixture. The method of calculating the composition is given:

Students should note that filtration of the mixture is a relatively slow process. Some students will note that the filtrate is not clear and colorless like distilled water. If the filtrate is not clear, consider refiltering to clarify the filtrate. The quality of the filter paper may dictate whether the filtrate is clear or not.

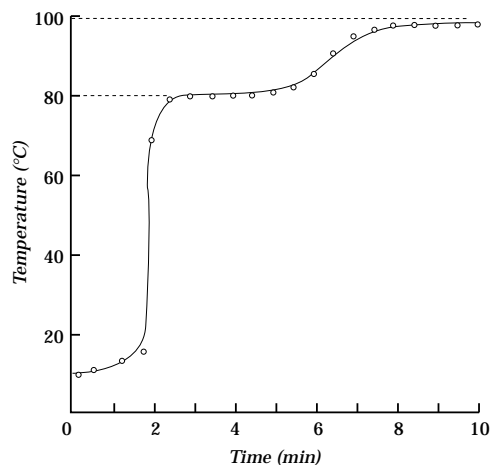


Figure 3. Typical student data for fractional distillation activity.

Part II. Fractional Distillation

Students will probably agree that it is impossible to tell that the original solution is a mixture of liquids. If heating is done slowly and carefully, students should obtain three separate fractions of liquid. Fraction 1 appears to be a pure substance having a boiling point of about 81 °C; Fraction 2 is a mixture of substances similar to the original solution; and Fraction 3 seems to be a pure substance with a boiling point the same as water. (Actually, if properly collected, Fraction 1 is a constant boiling azeotrope that is 90% alcohol and 10% water with a constant boiling point of 80.5 °C. It cannot be further separated by distillation, and thus it has the appearance of a single substance. Do not dwell on the purity of this fraction but emphasize that a separation has occurred.) The initial heating and subsequent graph of temperature *vs.* time is the key to determining the temperature ranges over which to collect the separate fractions (see Figure 3 for “typical” data).

There are two distinct plateaus or flat segments indicating that there are two different substances in the mixture, leading to the suggestion that three fractions should be collected to separate the two substances. Fraction 1 should be collected about 78-82 °C, Fraction 2 from 82 °C to

about the boiling temperature of water, and fraction 3 beginning at about the boiling point of water. (The boiling point of both liquids depends upon atmospheric pressure at time of the activity. Boiling points are lowered by a decrease in pressure.)

A typical set of properties determined for the solution and three fractions is shown in the following table.

Property	Solution	Fraction 1	Fraction 2	Fraction 3
Flammability	Yes	Yes	Yes	No
Odor	Medium	Strong	Medium	None
Density	~0.91 g/mL	~0.084 g/mL	~0.93 g/mL	~1.00 g/mL
Boiling point	Variable	~81 °C	Variable	100 °C
Dissolved sugar?	Yes	No	Slight	Yes

In order to obtain density data comparable to that listed above, students will need to make the mass and volume measurements carefully.

Part IV. Chromatography Procedure.

Students may not see actual separation of fractions on the column, but elution will allow them to see clearly three fractions, a yellow fraction absorbing at about 430 nm, a yellow-green fraction with absorption maxima at 460 and 600 nm and a green fraction with absorption maxima at 480 and 650 nm.

Answers to Implications and Applications

Part I. Gravity Filtration

1. Students will answer that gravity filtration is efficient at separating soluble and insoluble substances, but that it is not efficient from the standpoint of speed.
2. If the filtrate is unclear, some of the insoluble chalk passed through the filter paper. This is due to overfilling the funnel (so that some chalk slurry runs down between the filter paper and funnel), the filter paper is too porous to retain the smallest chalk particles, or to a hole punched in the filter paper.
3. By having the end of the funnel touch the side of the beaker, an unbroken liquid column is provided. This facilitates the flow and prevents spattering by having drops strike the liquid surface.

Part II. Fractional Distillation

1. Students should be able to conclude that a separation of two liquids has taken place on the basis of the evidence gathered by testing flammability, odor, and density of collected fractions.
2. On the basis of density, flammability, and odor tests, the first fraction was almost pure alcohol; the second fraction was quite similar to the original solution, and the third fraction was water.
3. If the fractions were redistilled, the following are the predicted results. Fraction 1 would boil at about 80 °C until it was all gone. Fraction 2 would come to a boil at about 80 °C and after all the alcohol was gone, the temperature would rise to the boiling point of water and remain there until all the liquid boiled away. Fraction 3 would begin to boil at about the boiling temperature of water and remain at that temperature as long as liquid remained.



Part III. Fractional Crystallization

1. The appearance of two solids and differences in solubility allow the conclusion that a separation has occurred.
2. Since the solutions are saturated at 10 °C, heating to 100 °C would not produce an observable change other than dissolving some of the undissolved solute present.
3. The sodium chloride and potassium nitrate may be separated by fractional crystallization because of the large difference in solubility at 100 °C and because the solubility of sodium chloride is nearly constant over the temperature range of 10 °C to 100 °C. When the hot solution is filtered, it contains almost pure potassium nitrate. Any excess sodium chloride remains in crystalline form. The filtrate, then, has little sodium chloride present, and when allowed to crystallize, the resulting solid is almost entirely potassium nitrate. If the solid is then put into solution and allowed to crystallize again, the residual sodium chloride remains in solution. Filtering and washing with water will result in removal of most of the sodium chloride.

Part IV. Column Chromatography

1. TTE removes rapidly the xanthophyll and carotenes. Chlorophyll and sodium carbonate strongly interact. Adding acetone, a polar solvent, reduces the interaction between chlorophyll and sodium carbonate by decreasing the number of adsorptive sites on the stationary phase (sodium carbonate). Chlorophyll A interacts with sodium carbonate less than does Chlorophyll B; hence, a smaller concentration of acetone is needed to help it move down the column.
2. A mixture like that in this activity may be separated in a chromatographic column because of different degrees of interaction of the pigments with sodium carbonate and the solvent.
3. Chromatographic separations are successful in many cases where other techniques fail due to the similarity in properties of the substances to be separated. The actinide oxides, for example, may be successfully separated in this way. These oxides could then be converted to the actinide element.

Post-Laboratory Discussion

Students will require minimal help with the data analysis for the simple filtration procedure. With fractional distillation, considerable help may be required, particularly in interpreting the results and deciding the probable composition of the fractions. Although the fractional crystallization and chromatographic separations are more complex to perform, students should be able to analyze their data with ease. Since it is unlikely that all of the procedures will be used with a class, several demonstrations are presented in the next activity section that may be used during the post laboratory period to show more than one separation technique. It is also recommended that other procedures be discussed at this time. For example, if students only do the filtration activity, then distillation, extraction, and/or chromatography activities may be demonstrated. Paper chromatography provides an interesting demonstration and is recommended regardless of which procedures are performed. Separations that were not done as part of the activity may be assigned as individual projects.

Extensions

Giving students the opportunity to perform one of the procedures not assigned to the entire class would be a suitable extension.

Additional laboratory activities involving separations may be found in *Laboratory Activity 1* in the *Forensic Chemistry* module. This activity uses thin layer chromatography (TLC) to separate the components in several over-the-counter drugs. This interesting laboratory activity uses a different chromatographic technique.

“Chalk Chromatography” in *Chemical Activities*, Teacher Edition (Borgford & Summerlin, 1988) is a unique chromatography activity with excellent instructions. (A student version of *Chemical Activities* is also available, but the teacher edition is sufficient if one is willing to type instructions for students.)

Assessing Laboratory Learning

1. Since all four procedures allow students to develop or practice laboratory techniques, one way to assess laboratory learning is to note which students are successful at performing the techniques involved. Hopefully, many students can successfully separate a mixture of soluble and insoluble substances by filtration.
2. On an examination, provide students with a graph showing the solubilities of several substances. Then ask students to use the data to select pairs of substances that might be separated by fractional crystallization. (To successfully use fractional crystallization, the substances to be separated should have *large* differences in solubility at *high* temperature. Secondly, the rate at which solubility changes with temperature should be quite different, *e.g.*, NaCl and KNO₃. Fractional crystallization has been used to separate NaCl, KCl, MgCl₂, and MgBr₂ from brines found in the Dead Sea between Jordan and Israel.)
3. What is one very important use of fractional distillation? (Fractional distillation is widely used in the petroleum industry to separate crude petroleum into several fractions containing useful hydrocarbon mixtures. Gasoline contains hydrocarbons that boil in a temperature range of 60-280 °C, jet fuel a range of 190-450 °C, kerosene a range of 350-550 °C, and so forth.)

CAUTION: Use appropriate safety guidelines in performing demonstrations.

Demonstration 1: Separation of Two Immiscible Liquids

Description

This demonstration illustrates the separation of immiscible liquids. It may be used when solutions and/or other mixtures are being considered.

Materials

Separatory funnel, 60- or 100-mL
Cooking oil, 50 mL
Distilled water, 50 mL, containing 1-3 drops of food coloring

Safety

After shaking the separatory funnel it is necessary to relieve the pressure buildup by inverting the funnel and opening the stopcock. Otherwise, the stopper could “blow.”

DEMONSTRATIONS



Procedure

Show students the two separate liquids. Pour them together into a large beaker. Ask students how the two could be separated completely. Pour the mixture from the beaker into a separatory funnel, stopper the funnel, and shake thoroughly. Relieve the pressure buildup. Set the funnel in a small iron ring attached to a ring stand and allow the liquids to separate. The oil layer will be the top layer. Remove stopper, then open the stopcock of the separatory funnel while holding it over the small beaker that originally contained the colored water and allow the bottom colored layer to drain out. Watch carefully so that the stopcock is closed at the precise moment when the water is completely drained (this may require some practice beforehand). Drain the remaining cooking oil into the small beaker that originally contained it. Show the class that the collected volumes are about the same as the original volumes. Figure 4 shows a sketch of a separatory funnel with the two immiscible liquids.

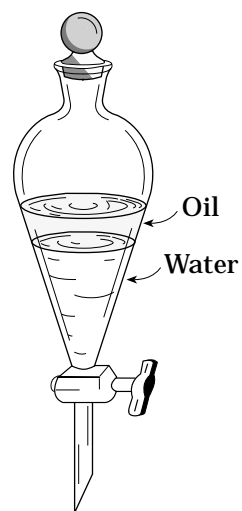


Figure 4. Separatory funnel.

Remarks

About 10 min preparation time to try the demonstration prior to performing it for students is required. 15-20 min are needed to perform the demonstration along with an explanation of what is occurring. Have colored water prepared in advance. A graduated beaker is sufficiently accurate for measuring liquid volumes. Thoroughly shake the two liquids together in the funnel while the class observes. Students may be called upon to help.

Little discussion is needed; the observations tell the story. It might be wise to indicate how the demonstration fits into the topic being considered. Ask students why the oil comprises the top layer of the two layers in the funnel [*less dense*]. How do they know it is the oil?

Demonstration 2: Fractional Distillation of Two Miscible Liquids

Description

This demonstration may be used if the fractional distillation activity is not selected for student use.

Materials

Thermometer
Distilling flask, 250-mL
Boiling chips or glass beads
Condenser
Ringstand, utility clamps, iron ring
Ceramic-centered wire gauze
Hot plate
Methanol, 50 mL
Distilled water, 50 mL
Safety goggles

Safety

Wear safety goggles and use a hot plate rather than an open flame. Methanol is both toxic and highly flammable.

Procedure

Mix 50 mL methanol with 50 mL distilled water. Pour the mixture into a 250-mL distilling flask and set up the distilling apparatus as illustrated in Figure 1 of *Activity 1, Part II. Fractional Distillation*. Check fittings in distillation apparatus for tightness since methanol vapors are very flammable. Heat the mixture at a temperature below the boiling point of water (100 °C/1 atm), but above the boiling point of methanol (65 °C/1 atm). The alcohol will distill first.

Remarks

See *Activity 1, Part II. Fractional Distillation* for details concerning the apparatus and procedure.

Demonstration 3: Separation of Chlorophyll Pigments by Paper Chromatography

Description

This demonstration illustrates separation of pigments by paper chromatography.

Materials

Fresh or frozen spinach (or green ivy)
Mortar and pestle
Washed sand
Solvent mixture (25 mL hexane, 25 mL ethanol, and 2 mL 6 M acetic acid)
Coffee filter paper strip about 1 inch by 8 inches (regular filter paper may be used)
Large test-tube (25- x 200-mm)
Cork to fit test-tube
Paper clip

Safety

Since the solvent mixture is highly flammable, do not have open flames nearby.

Procedure

Prepare a mixture of 25 mL hexane, 25 mL ethanol, and 2 mL 6 M acetic acid (1.7 mL conc reagent/ 5 mL solution). Grind a handful of spinach or green ivy with sand in a mortar and pestle in 25 mL of the prepared solvent. The pigments in the cells will dissolve into the solvent, giving a dark green liquid.

Pour 10 mL fresh solvent mixture into a large test-tube and place in a test-tube rack. Cut a piece of coffee filter paper about one inch wide and long enough to reach the bottom of the test-tube. Dip a pipet into the green mixture and carefully spot the pigment onto the filter paper about one inch from the end of the paper. Straighten a paper clip with one end resembling a hook. Insert the straight end into the bottom of a cork stopper and hang the strip of filter paper from the hook, the pigment dot on the end away from the hook. The pigment spot on the filter paper should be as small as possible. It may be necessary to spot several times to build up the concentration of pigment. Allow the spot to dry between applications. This might be done prior to class.

Insert the cork stopper into the test-tube so the end of the filter paper just barely touches the solvent. (*NOTE: The pigment dot must be above the level of the solvent.*) Set upright and observe the separation of the chlorophyll pigments. The same procedure may be done substituting different types of pen inks. Washable inks, particularly black, work well.

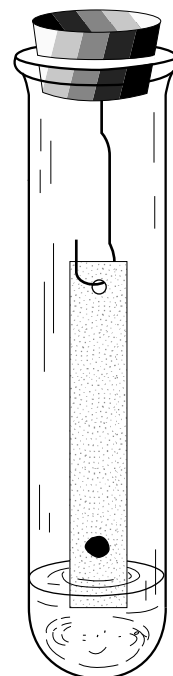


Figure 5. Paper chromatography set up.



Remarks

This demonstration may be used anywhere chromatographic separation fits in. If students do not perform a paper chromatography procedure, then the demonstration should be done.

The time needed depends upon the method used. If students help with grinding the spinach leaves and other tasks, then the demonstration will take most of a period. If you do the entire demonstration, then the entire period will be needed. In either case, the demonstration should be rehearsed prior to presenting it to a class.

NOTE: Bob Becker (Kirkwood High School, Kirkwood, MO) suggests a circular chromatography demonstration or activity using water as a solvent and Vis-a-Vis™ washable overhead projector pens. Make a pinhole in the center of a 12-cm diameter filter paper circle, fit with a cone-shaped wick of filter paper that penetrates the pinhole, and spot four colors (blue, black, red, and green are most effective) at about 1 cm from the central pinhole. Place the assembly in a Petri dish with a slightly smaller diameter than the filter paper that has about 1/2 cm of water in the bottom. Allow the water to wick up the cone-shaped central wick and chromatograph the four spots. A lovely separation pattern appears. Alternatively, you can use only a black marker in a ring. If you collect pens from several years, you will note that each has a “tag” from that year; you can ask students to see if one can distinguish between the pens from year to year.

GROUP AND DISCUSSION ACTIVITIES

Key Questions

1. What are the differences in properties that may be used to separate the components of a mixture? *[Differences in boiling point, particle size, solubility, densities, magnetic properties, volatility.]*
2. Name some techniques used to separate the components of a mixture. *[Simple distillation, fractional distillation, filtration, crystallization and fractional crystallization, attraction to a magnet, paper chromatography, thin layer chromatography (TLC), gas chromatography, column chromatography, high performance liquid chromatography (HPLC), gel electrophoresis.]*
3. What purpose is served in separating the components of mixtures? *[Separating the components of a mixture may serve several purposes. In order to use many of the modern instruments available (spectrometers, etc.), it is frequently necessary to have pure substances. Many analytical procedures depend upon separating mixtures to identify the components. Separation of undesired contaminants is important in the preparation of chemical products. Petroleum refining is based upon first separating the crude petroleum into different fractions (fractional distillation). The preparation of many biologically active substances requires careful separation. For example, some biologically active substances exist as enantiomers (lefthand and righthand stereoisomers), of which only one isomer is the active form. What students give as an answer will depend upon their knowledge. You will probably need to help them.]*
4. How do components of a mixture affect the properties of a mixture? *[Mixtures generally have properties that differ from those of the pure components. Some properties that are different for mixtures are melting point, boiling point, volatility, density, physiological activity, reactivity, and so forth.]*

5. How does a mixture differ from a compound?*[A mixture may be either heterogeneous or homogeneous. In either case, the mixture does not have a definite composition. For example, concentration of salt dissolved in water can have an infinite number of values. Compounds always have a definite composition as generalized by the law of definite proportions. Mixtures may be separated into their components by physical methods whereas compounds cannot.]*
6. How does a homogeneous mixture differ from a heterogeneous mixture? *[A homogeneous mixture has a uniform composition but a heterogeneous mixture does not. For example, a 1-mL sample of a 10% salt solution has the same composition regardless of where in the bulk solution a sample is obtained. Granite, on the other hand, illustrates a heterogeneous mixture. One sample of granite will have a different composition by mass from that of another sample of granite from the same source.]*
7. What are some household products that are the result of separation techniques?*[Table sugar is produced by the crystallization of a solution. Salt may be refined to remove undesired impurities, and it can be prepared by the evaporation of brine. Distilled vinegar is prepared by fractional distillation. Flavorings used in baking, candy making, beverages, perfumes, etc. are obtained by extraction. For example, fragrances used in perfumery are extracted from natural sources. Vanilla is extracted from vanilla beans using an alcohol as the extracting solvent. Decaffeinated coffee has the caffeine extracted by either a chemical process or by a “natural” process. The chemical process uses methylene chloride as the extracting solvent while the “natural” process uses water as the extracting solvent. Methylene chloride is highly toxic and a known carcinogen.]*

Extensions

A suitable activity for students is to observe labels on common household products and over the counter drugs for information about purity. (If students are sent to the store, talk to the store manager prior to sending students there to take notes.) Products to be checked include household cleansers, household ammonia, baking soda, rubbing alcohol, moth balls and moth crystals, food products such as salad oil, common analgesics such as aspirin, etc.

An alternate activity is to have students bring products from home or to survey the products that are present in homes. In particular, students might pay attention to such things as paint thinner, paint remover, pesticides, herbicides, etc. It is not recommended that pesticides, herbicides, flammable solvents, and toxic substances be brought to school. Students can copy the needed information from the labels.

Pictures in the Mind

Gas-Liquid Chromatography (GLC)

Gas-liquid chromatography is used to separate mixtures of gases and/or volatile liquids. The mixture is first vaporized and then passed through a long, narrow column packed with some finely divided solid that is coated with a nonvolatile liquid. A carrier gas, usually helium or nitrogen, sweeps the vaporized mixture through the column. The mixture vapors move along the column at different rates since they interact to different degrees with the column's liquid coating. The vapors that interact the least travel the fastest through the column while those that interact most strongly travel the slowest.



The result is that the fastest vapors exit the column first; the slowest exit last. In this way the components of the mixture are separated. At the exit end of the column, a detector detects the vapor's passing and sends along the information to a recording or memory device. When the information is displayed on a moving piece of chart paper or on a computer monitor screen, it is called a chromatogram. In this way chemists are able to "see" the number of components in a mixture.

Gas chromatography has been developed to the extent that it is possible to separate extremely complex mixtures when only nanogram (10^{-9} g) quantities are available. This development combined with advances in detectors makes the detection and analysis of trace quantities in the picogram (10^{-12} g) level possible. It is even possible to separate compounds that are different only in their isotopic composition.

A way of picturing the gas chromatography process is to imagine a large flock of seagulls (molecules) traveling down a coastline. While all are wafted along by the same tailwind (carrier gas), some stop off to feed on the ocean's (stationary phase) riches, and arrive at a given geographic point (detector) later than those gulls that did not stop or stopped only occasionally along the way. Molecules differ from seagulls in this analogy in one important respect: Chemically identical molecules ideally will behave identically with respect to the stationary phase, thus arriving at the destination (detector) in distinct clumps, or peaks.

While this analogy is simplistic, it helps visualize the chromatographic process.

Counterintuitive Examples

How pure is pure? If orange juice (pure orange juice) is filtered to remove the pulp, is it pure or a mixture? Is gasoline pure? Table salt? Iodized salt? Ivory soap—what does "99 and 44/100ths % pure" mean?

Ivory Soap is advertised to be "99 and 44/100ths percent pure." Does this mean the soap really contains 99.44 alkali metal salts of fatty acids out of every 100 "molecules"? A statement in the *Content in a Nutshell* section says that nothing is absolutely pure. So this statement about Ivory Soap means the limits of purity must be definable and measurable. In the case of Ivory Soap, when compared to three expensive imported soaps, it was found to contain fewer impurities. That is where the sales angle of "99 and 44/100ths percent pure" originated. The air mixed with the soap that lets it float is an impurity.

Most people are familiar with the "sweet" smell of air after a thunderstorm or rain. The falling water seems to remove all undesirable substances. Is it pure? Air is a mixture and not a pure substance.

Pure orange juice is another case in point. It too is a mixture although it may be made only from fresh oranges. In fact, no substance is completely pure. Impurities are always present. The impurities may be from the atmosphere, from unremoved processing materials, or even from the container holding the "pure" substance.

Modern techniques make it possible to detect impurities in substances that may have been previously considered pure. Parts per million (ppm), parts per billion (ppb), and even parts per trillion (ppt) detection has been achieved. Units of mass, molecules, or volume may be used. For example, an impurity present at 1 ppm by mass means that 1 g of material contains 1 millionth of one gram of impurity. This is 1×10^{-6} g (1 μ g) in the 1 g sample. Here is where separation techniques play an important part.

An area of active research is connected with separation techniques that allow reliable and rapid analysis of complex mixtures of pollutants found in toxic wastes, polluted streams and lakes, and contaminated biological samples. Analytical methods allowing the separation and quantitative measurement of each of the 22 individual isomers of tetrachlorodioxin at the parts per trillion range (one part in 10^{12} or 1×10^{-12} g in a 1 g sample) have been done. With such advanced techniques it is possible to pinpoint the impurities in practically any substance.

Other

Separations, instrumentation, and environmental quality – Chemistry in action.

According to *Opportunities in Chemistry Today and Tomorrow* (Pimentel & Coonrod, 1987), three chemically related questions are asked:

- “What potentially undesirable substances are present in our air, water, soil, and food?”
- “Where did these substances come from?”
- “What options are there—alternate products and processes—to reduce or remove known problems?”

Determining what substances are present in the environment requires analytical chemists to act as detectives. To do so they need to develop sensitive and selective analytical techniques. Analytical chemists deal in separations and instrumentation. Determining where the substances originate calls for the action of analytical chemists along with collaboration by meteorologists, oceanographers, geologists, climatologists, biologists, and hydrologists. The development of alternatives, however, requires that problems be tackled by the entire society.

Detection may be protection. A point that must be made is that simply detecting the presence of a substance does not mean it is a hazard. Chemists need to use their skills at detecting minute concentrations well below the hazard threshold so that frantic corrective action need not be taken. Although it seems trite, proaction rather than reaction is needed. For example, carbon monoxide is dangerously toxic at concentrations greater than 1000 ppm. This compound is always present in the atmosphere at about 1 ppm, and prolonged exposure to concentrations greater than 10 ppm is considered to produce negative health effects. Should CO be removed completely from the atmosphere? That would probably be impossible and prohibitively expensive. The real need is to be able to decide where cleanup should begin between the safe range and the known toxic threshold. This decision requires the ability to separate the CO from the other atmospheric constituents, or to develop effective instrumentation to measure it in place, thus providing protection through detection.

Some people have called for a “zero risk” approach to the environment, meaning that every single molecule of CO would be removed from the atmosphere. This goal is clearly unattainable from both scientific and economic points of view. This trend has, fortunately, been gradually replaced by a sophisticated risk assessment/risk management approach. In this approach costly crash projects are replaced by long-term fundamental environmental monitoring. Continuous monitoring requires good analytical techniques. These techniques, in turn, require improved sensitivity, selectivity, separation, sampling, accuracy, speed, and data interpretation. Separations, instrumentation, and computers are key players in such an effort.



Analytical methods to separate and measure quantitatively the 22 different isomers of tetrachlorodioxin at the parts per trillion level (1 part in 10^{12}) have been developed. The concentration of nitric acid and formaldehyde in Los Angeles smog can now be measured by infrared spectroscopy. The determination measures successfully the simultaneous concentrations of formaldehyde, nitric acid, peroxyacetyl nitrate, and ozone in the air at the parts per billion range. Although one part per billion may be too small to pose a direct hazard, its presence can be significant in atmospheric chemistry. Sulfur dioxide concentration at the ppm level in the smoke plumes of power plants has been measured. It is also possible to detect the pollutants directly from the exhaust pipe of an internal combustion engine, as in automobile exhaust.

TIPS FOR THE TEACHER

Language of Chemistry

azeotrope any mixed liquid with a boiling point lower than any liquids making up the mixture.

distillation purification process in which a liquid is evaporated and then condensed again to a liquid; used to separate dissolved solids from liquids or liquids from liquids according to boiling point.

electrophoresis movement of charged particles in a liquid or a gel caused by an electromotive force derived from a voltage applied to electrodes in contact with the liquid or gel.

filtrate liquid that passes through the filtering apparatus when a solid-liquid mixture is filtered.

filtration process used to separate an undissolved solid from a liquid.

fractional crystallization separation of two solids by using a difference in solubility at relatively low temperature; also called recrystallization.

fractional distillation separation of miscible liquids having different boiling points by heating, condensing the vapor, and collecting the distillate in fractions according to boiling point.

mixture combination of two or more substances not chemically combined and without definite proportions.

percent composition percent by mass of each component in a mixture.

polarity charge displacement in a molecule due to the different electronegativities of the component atoms, *e.g.*, polarity of water.

precipitation formation of a solid from components previously in solution.

purity degree to which a homogeneous substance consists of only one component.

residue solid that is retained in the filter paper during filtration.

solubility extent to which a solute dissolves in a solvent.

solute dissolved substance in a solution, *i.e.*, the dispersed component.

solution homogeneous mixture consisting of two or more components.

solvent dispersing or dissolving medium in a solution; generally present in greater quantity than the solute.

Pattern Recognition

Like dissolves like—polar molecules dissolve in polar solvents and nonpolar molecules dissolve in nonpolar solvents; a general rule that is probably overused since there are a number of exceptions.

Common Student Misconceptions

“Pure aspirin’ (or other product labeled ‘pure’) contains only aspirin.”

Although common products are frequently labeled as being pure, in most cases they are not. Aspirin is made with pure aspirin, but the aspirin tablet is primarily filler (starch or other digestible substance) with 325 mg of sodium acetylsalicylate. The same is true of other analgesics such as ibuprofen (Advil[®]) and acetaminophen (Tylenol[®]).

Decision Making

Information about decision making as it pertains to both separations and instrumentation is presented in *Decision Making* in the *Instrumentation* module.

Chromatography is a process based on differences in the rate at which the components of a mixture move through a stationary phase while under the influence of a moving phase. The word “chromatography” comes from the Greek words for “color” (khroma) and “to write” (graphein). Chromatography results in “color writing.” The Russian botanist, Mikhail Tswett (1872-1920), is considered to be the first to have described how to separate mixtures of plant pigments using column chromatography. Paper chromatography was considered so important in the scientific world that its developers, Martin and Synge, received the Nobel Prize in 1952.

HISTORY: ON THE HUMAN SIDE

1. Word Search (see *Appendix* for master copy)

N H U L M R W K G O X A G F U N H
 O N H I G A N O I T U L O S G I H
 I Q V F I S P A I T J S I J L S M
 T Z U E R X D L J H S V S O T T I
 A M C D R A I E P O R T O E Z A L
 L G R C A B C B A L E Z T S P Y L
 L R F Z U V F T B G W U G U M L I
 I P E L S Y J V I S L A Q C O E O
 T Y O X G H U X D O P M J U J D N
 S S N U O I X P S E N U E M L T F
 I C R Y S T A L L I Z A T I O N V
 D U J E W C H E T A R T N I F R M
 X Z L Y H P A R G O T A M O R H C

HUMOR: ON THE FUN SIDE

Words about the concepts in this module can be obtained from the clues given. Find these words in the block of letters:

1. Type of distillation that separates miscible liquids.
2. Liquid that passes through filter paper when filtering a solid-liquid mixture.
3. The dissolved substance in a solution.
4. Separation of a solid from solution based on a difference in solubility at low temperature.
5. Process of evaporation and condensation in a separate vessel.
6. Technique used to separate colored substances in a green leaf extract.



7. A mixture of liquids with a boiling point lower than any liquid in the mixture.
8. Extent to which a solute dissolves in a solvent.
9. Homogeneous mixture of two or more components.
10. The "m" in ppm.

Answers: 1. FRACTIONAL 2. FILTRATE 3. SOLUTE
4. CRYSTALLIZATION 5. DISTILLATION 6. CHROMATOGRAPHY
7. AZEOTROPE 8. SOLUBILITY 9. SOLUTION 10. MILLION

2. See cartoons at end of module.

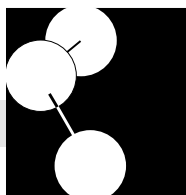
MEDIA

1. *The World of Chemistry* videotape "Number 22: Polymers." World of Chemistry Videocassettes. Annenberg/CPB Project, P.O. Box 1922, Santa Barbara, CA 93116-1922; (800) 532-7637; World of Chemistry Series, Atlantic Video, 150 South Gordon Street, Alexandria, VA 22304; (703) 823-2800 or QUEUE Educational Video, 338 Commerce Drive, Fairfield, CT 06430; (800) 232-2224.
2. Software published by *JCE: Software*, a publication of the *Journal of Chemical Education*, Department of Chemistry, University of Wisconsin-Madison, 1101 University Avenue, Madison, WI 53706-1396: (608) 262-5153 (voice) or (608) 262-0381 (FAX).

Viscosity of Polymer Solutions, by Gary L. Bertrand. Vol. V A, No. 2, for the Apple II computer.
3. Software published by Project SERAPHIM, Department of Chemistry, University of Wisconsin-Madison, 1101 University Avenue. Madison, WI 53706-1396: (608) 263-2837 (voice) or (608) 262-0381 (FAX).
 - a. For the Apple II computer running on ProDOS: AR 701
 - b. For the Apple II computer: AP 702, AP 705, AP 209
 - c. For IBM PCs and PC-compatibles: PC 3601, PC 4901
4. Videodiscs published by *JCE: Software*, a publication of the *Journal of Chemical Education*, Department of Chemistry, University of Wisconsin-Madison, 1101 University Avenue, Madison, WI 53706-1396: (608) 262-5153 (voice) or (608) 262-0381 (FAX).
 - a. "From an Amino Acid to a Peptide Chain," "The Alpha Helix," "Hair and Silk," and "DNA Structure, Synthesis of Messenger RNA, Protein Synthesis," four chapters on *The World of Chemistry: Selected Demonstrations and Animations: Disc II* (double sided, 60 min.), Special Issue 4.
 - b. "Polymers," "Carbohydrates," and "Proteins," three chapters on the videodisc *Demonstrations in Organic Chemistry* (double sided, 60 min.), Special Issue 6.
5. *Elements, Compounds and Mixtures*, 20 min. video available from Coronet/NTI Film and Video, 108 Wilmot Road, Deerfield, IL 60015; (800) 621-2131; (708) 940-3640 (FAX).

EQUIPMENT AND INSTRUMENTATION

Glassware, such as adapters, condensers, funnels, flasks, beakers, *etc.* used in the procedures in this module, is available from most scientific supply houses, like Sargent-Welch, Cenco, EMD Fisher, Flinn Scientific, *etc.* This is also true of chemical substances.

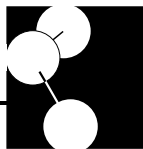


Links/Connections

The topic relates separation of components of mixtures to different fields of chemistry.

WITHIN CHEMISTRY

1. Separation techniques are related to chemical and physical properties, states of matter, and phase changes. Separations are the basis for many laboratory experiences. Data analysis involves the use of mathematics. A knowledge of properties of mixtures as well as pure substances is required in designing separation techniques.
2. Industrial chemistry uses separations in quality control and monitoring of reaction streams as well as in batch and continuous processes in the manufacture of chemicals.
3. The petroleum industry is based on separation of components of crude oil by fractional distillation. A visit to a local petroleum refinery could be an excellent field trip for a chemistry class.
4. A helium refinery is another example of industrial separation of components of a mixture. Helium occurs in high percentages in natural gas deposits in the Texas panhandle near Amarillo. This is the only such refinery in the United States. Separation of helium from the natural gas involves compressing the gas mixture until the methane and other gases liquefy, leaving only helium as a gas. The helium is bled off, purified, and processed further by carefully controlling the pressure above the liquefied gas. The methane is returned to the well from which it was taken or sold outright. The concept of critical pressure and critical temperature can be applied to this technique.
5. Forensic chemistry utilizes separation techniques in determination of concentrations of various substances in the bodies of victims of crime or a person suspected of committing a crime. Illegal drugs, alcohol, and other life-threatening toxic substances are subject to investigation and require separation from other substances.
6. Medical technology laboratories in hospitals and the private sector are involved in separation techniques to determine concentrations of various substances in body tissues and body fluids. Detection of various inheritable diseases (diabetes), infectious diseases (hepatitis), and degree of injury due to a heart attack are examples of biomedical analyses that depend upon separation and instrumentation.
7. The weather bureau actually does separation techniques in determining the pollen count of the air each day.
8. Gaseous effusion was used to separate the different isotopes of naturally occurring uranium as gaseous uranium hexafluoride. By allowing the UF_6 to effuse through a series of effusion orifices, the lighter ^{235}U isotope was successfully separated from the heavier ^{238}U isotope.
9. Electrophoresis is another recent development (after 1930). In this technique ions (electrically charged particles) move through a liquid caused by an emf (developed by a voltage source) applied to electrodes in contact with the suspension or solution. It is particularly useful in separating a mixture of proteins or the components of blood serum. It is also useful in separating DNA and RNA fragments.



10. Ultracentrifugation is another separation process that is widely used in biochemistry. In this process a mixture is placed under the influence of a centrifugal force that is many times greater than the earth's gravitational field, causing a different rate of sedimentation of different molar mass substances. Ultracentrifuges spin at rates of 5×10^4 revolutions per minute or more.
11. In order to separate the sulfur dioxide (SO_2) from the combustion products derived from burning fossil fuels, the effluent gases are passed through a piece of equipment called a scrubber. In this device, the gases are sprayed with a water suspension of lime (CaO), which converts the gaseous sulfur dioxide to solid calcium sulfite (CaSO_3) and calcium sulfate (CaSO_4).

BETWEEN CHEMISTRY AND OTHER DISCIPLINES

Since the distinction between chemistry and other disciplines, including the arts and social sciences, is rapidly disappearing, the links between chemical uses of separation and uses in other disciplines is rather broad. Medicine, in particular, makes wide use of separations in microbiology, histology, and pharmacology. (Some medical uses of separations techniques were described in *Within Chemistry*.)

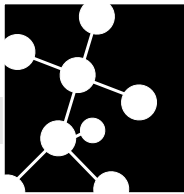
TO THE CONTEMPORARY WORLD

All of the separation techniques discussed in this module are currently used by the chemical industry, in medical research, in routine medical testing, in pollution monitoring and abatement, by many agencies in the governmental and private sectors, and others. These techniques now are in widespread use and will remain so.

1. **Careers.** Analytical chemist, soil analysis, technician, medicine, environmental chemist, forensics medical examiner, medical technology, water quality control, biochemist, field biologist, petroleum engineering, teacher-professor.
2. **Community.** As mentioned previously, separation is used in many endeavors that are present and operational in most communities throughout this country as well as much of the world.
3. **Field trips.** Petroleum refinery, private analytical laboratories, water quality control board, criminology laboratories, forensics laboratory, sewer plant, metal plating industries, helium refinery, dairy industry, hospital laboratory, mining sites, pharmaceutical industries.
4. **Knowledgeable individuals.** Analytical chemists in industrial and private sectors, physicians, soil chemists, university professors, pharmacologists, forensic chemists, medical examiners, dairy chemists, environmental chemists and biologists, local water quality chemist.

SOCIETAL (SCIENCE/ TECHNOLOGY/ SOCIETY; CURRENT EVENTS)

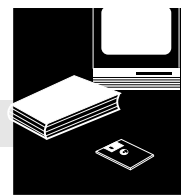
See *Instrumentation* module.



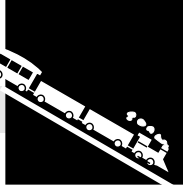
Extensions

Students can prepare poster sessions to be presented to the class. A poster session may include any facet of solution chemistry and separation technique employed in the private sector, industry, medicine, *etc.* The session includes poster boards illustrating the information. The presentation may last for twenty to thirty minutes. Information may be obtained from interviews, field trips, personal research, or library research.

References

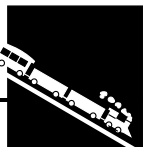


- Module developed by Royace Aikin and Daniel Kallus, the Texas team.
- Borgford, C. L., and Summerlin, L. R. (1988). *Chemical activities*. Washington, DC: American Chemical Society.
- Excellent compendium of simple, easy to perform activities. Especially recommended for this module are some of the activities in the section, *Chemical Detectives: Tools and Techniques of the Chemist*. "Chalk Chromatography" is a particularly interesting activity.
- Budavari, S. (Ed.). (1989). *The Merck index* (11th Ed.). Rahway, NJ: Merck and Co.
- Excellent general reference for properties, preparation, and hazards of chemicals. It also contains many useful tables in the appendix along with a formula index and cross index.
- Lippincott, W. T., Gailey, K. D., Meek, D. W., and Whitten, K. W. (1984). *Experimental general chemistry*. Philadelphia, PA: Saunders.
- Excellent laboratory manual that may well serve as a general reference for general laboratory procedures and instrumental techniques. The lab manual may also be used to select particular parts of experiments to use with high school classes.
- McQuarrie, D. A., and Rock, P. A. (1984). *General chemistry*. New York, NY: Freeman.
- This book is an excellent general chemistry reference for the teacher. It contains extensive coverage of the same topics taught in a typical high school chemistry course, but in greater depth.
- Parry, R. W., Bassow, H., and Merrill, P. (1987). *Chemistry: Experimental foundations* (4th Ed.). Englewood Cliffs, NJ: Prentice-Hall.
- The only CHEMS-derived text currently published. Very good information, especially how to present chemistry from an experimental point of view. The companion teacher's guide is a must.
- Pimentel, G. C. (Ed.). (1963). *Chemistry: An experimental science*. San Francisco, CA: Chemical Education Material Study and W. H. Freeman.
- Although it is now out of date and perhaps out of print, it still is a good reference for "seeing" how to present chemistry through "discovery." The accompanying teacher's guide is useful as are many of the experiments in the laboratory manual.
- Pimentel, G. C., and Coonrod, J. A. (1987). *Opportunities in chemistry today and tomorrow*. Washington, DC: National Academy Press.
- Recommended reading for all high school chemistry teachers.
- Summerlin, L. R., Borgford, C. L., and Ealy, J. B. (1987). *Chemical demonstrations: A sourcebook for teachers* (Vol. 2). Washington, DC: American Chemical Society.
- The demonstrations, "Colorful Mixture Separations" and "Separating Metallic Iron from Cereal" are suitable for this module (see *Food and Chemistry* module). Many of the other demonstrations are useful in any introductory chemistry course.
- Wilbraham, A. C., Staley, D. D., Simpson, C. J., and Matta, M. S. (1987). *Chemistry laboratory manual*. Menlo Park, CA: Addison-Wesley.
- A good, solid high school chemistry laboratory manual, although in a somewhat cookbook style. It presents useful experiments including a good presentation of laboratory safety and laboratory techniques.

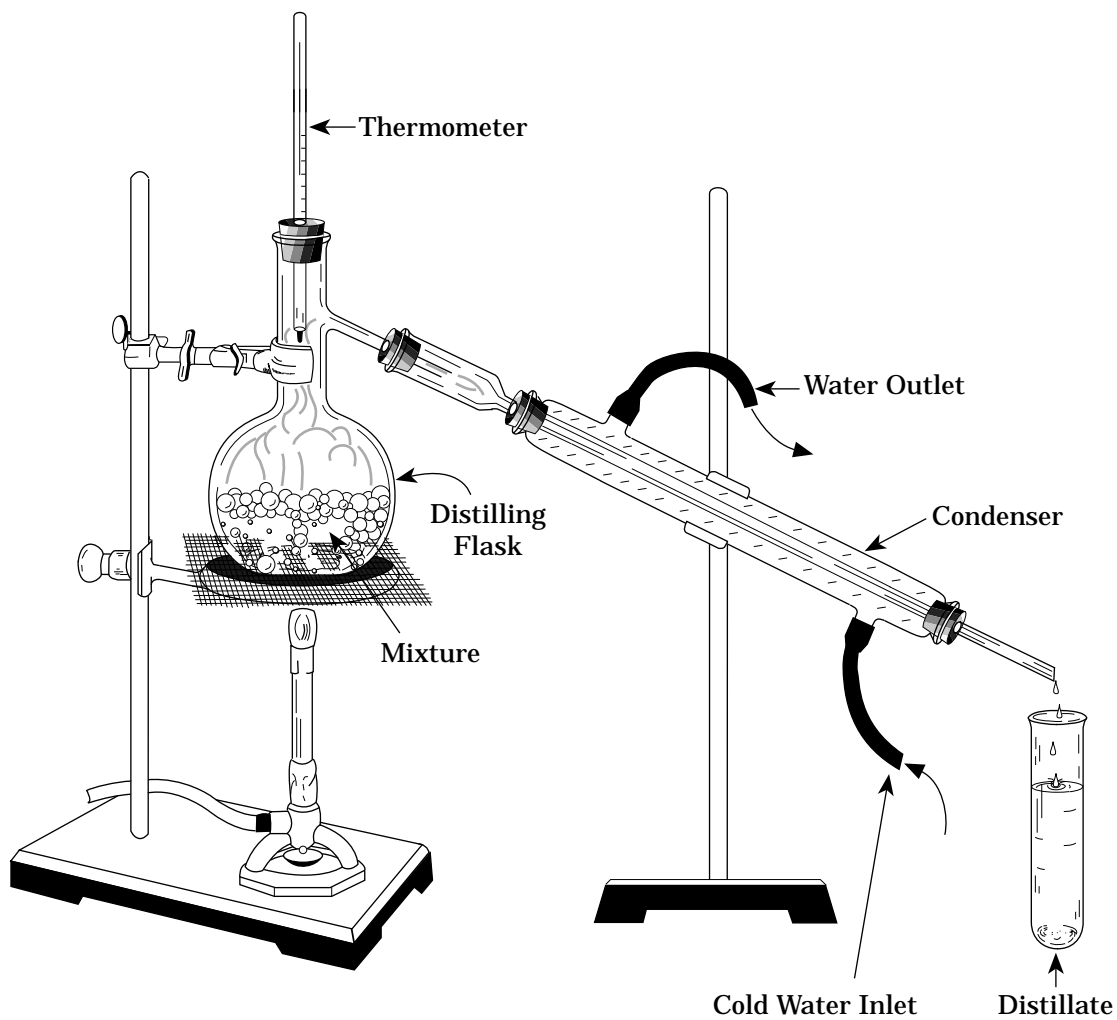


Appendix

- **Transparency Masters**
 1. A Simple Distillation Apparatus
 2. Typical Apparatus Used in Separation Techniques
 3. Word Search
- **Humor**

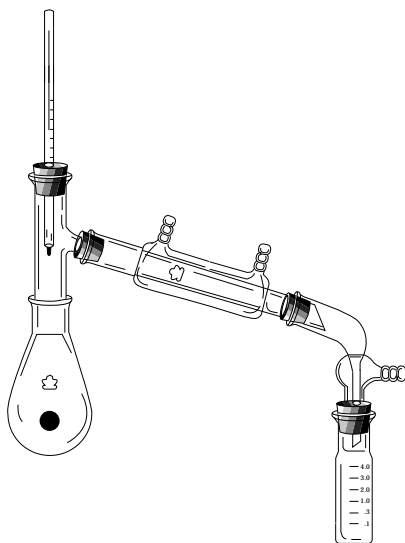
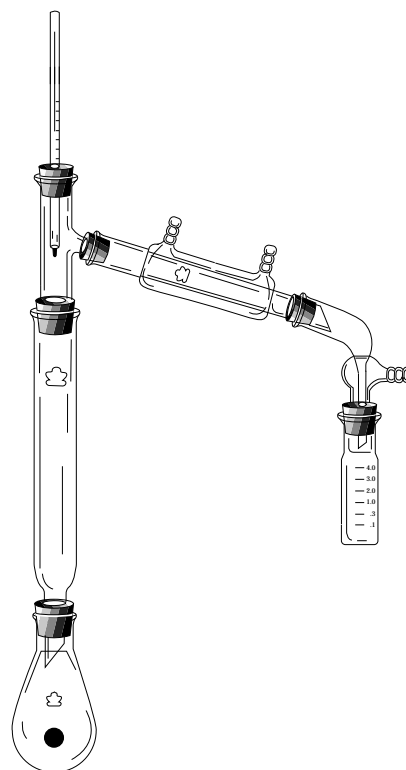
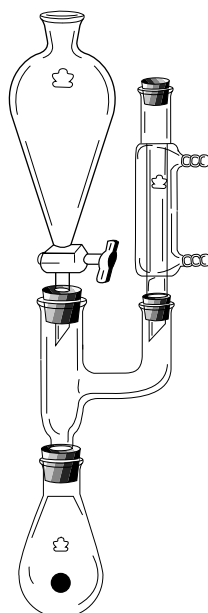


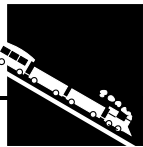
A Simple Distillation Apparatus



Typical Apparatus Used in Separation Techniques

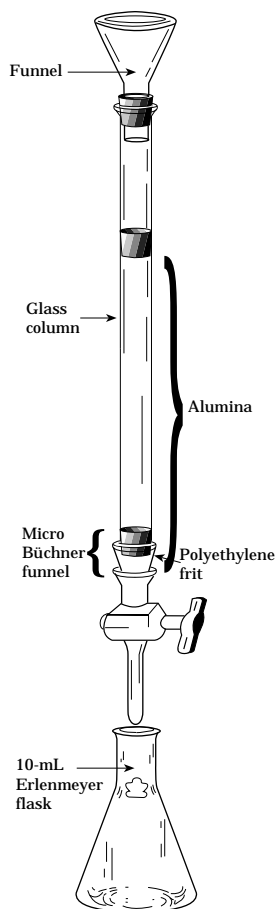
(Courtesy of Kontes Glass Company)

Simple Distillation**Fractional Distillation****Reflux with Addition**

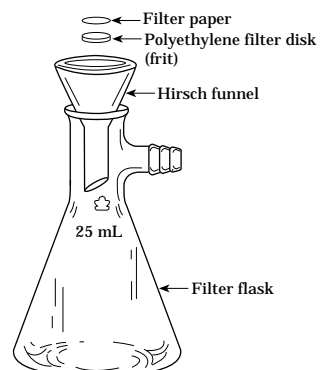


Typical Apparatus Used in Separation Techniques Cont.

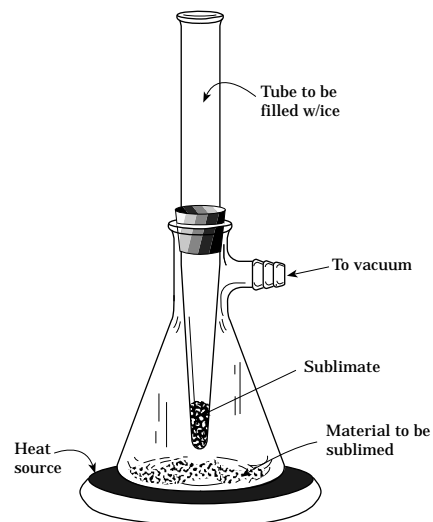
Column Chromatography



Hirsch Funnel Filtration



Sublimation

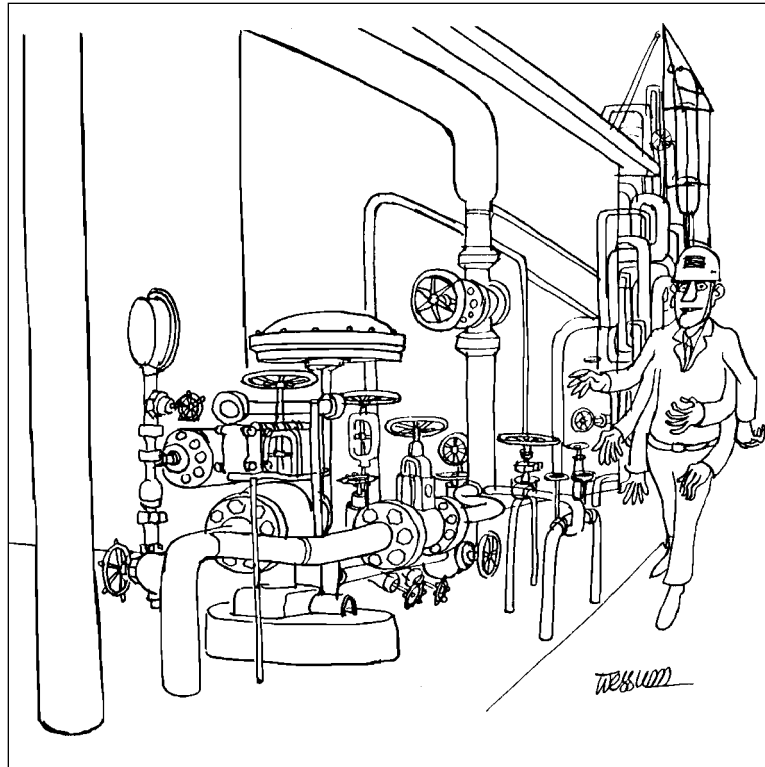
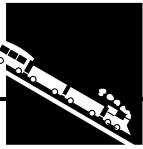


Word Search

N H U L M R W K G O Y A G F U N H
O N H I G A N O I T U L O S G I H
I Q V F I S P A I T J S I J L S M
T Z U E R X D L J H S V S O T T I
A M C D R A I E P O R T O E Z A L
L G R C A B C B A L E Z T S P Y L
L R F Z U V F T B G W U G U M L I
I P E L S Y J V I S L A Q C O E O
T Y O X G H U X D O P M J U J D N
S S N U O I X P S E N U E M L T F
I C R Y S T A L L I Z A T I O N V
D U J E W C H E T A R T L I F R M
X Z L Y H P A R G O T A M O R H C

Words about the concepts in this module can be obtained from the clues given.
Find these words in the block of letters:

1. Type of distillation that separates miscible liquids.
2. Liquid that passes through filter paper when filtering a solid-liquid mixture.
3. The dissolved substance in a solution.
4. Separation of a solid from solution based on a difference in solubility at low temperature.
5. Process of evaporation and condensation in a separate vessel.
6. Technique used to separate colored substances in a green leaf extract.
7. A mixture of liquids with a boiling point lower than any liquid in the mixture.
8. Extent to which a solute dissolves in a solvent.
9. Homogeneous mixture of two or more components.
10. The "m" in ppm.



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THE WIZARD OF ID

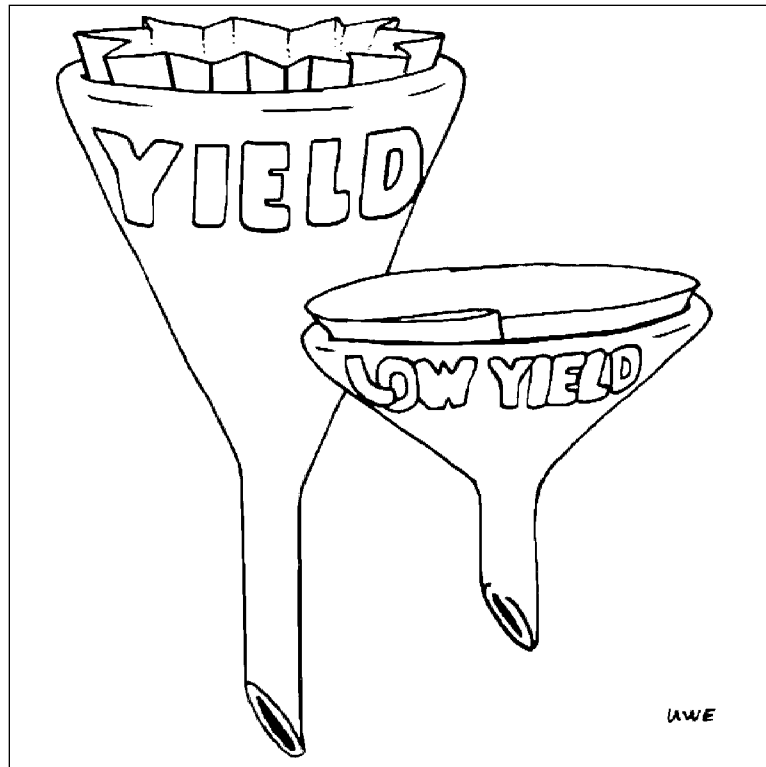
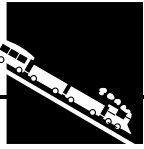
Brant parker and Johnny hart



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CHEM 13 NEWS, April 1982, p. 5. Reprinted with permission.