Experiment

Separation of the Components of a Mixture

OBJECTIVE

To become familiar with the methods of separating substances from one another using decantation, extraction, and sublimation techniques.

APPARATUS AND CHEMICALS

Apparatus

balance
Bunsen burner and hose
tongs
evaporating dishes (2)
watch glass
50 or 100 mL graduated cylinder
clay triangles (2) or wire gauze (2)
ring stands (2)
ion rings (2)
glass stirring rods

Chemicals

unknown mixture of sodium chloride, ammonium chloride, and silicon dioxide

DISCUSSION

Most of the matter people encounter in everyday life consists of mixtures of different substances. Mixtures are combinations of two or more substances in which each substance retains its own chemical identity and therefore its own properties. Whereas pure substances have fixed compositions, the composition of mixtures can vary (☞ Section 1.2). For example, a glass of sweetened tea may contain a little or a lot of sweetener. The substances making up a mixture are called *components*. Mixtures such as cement, wood, rocks, and soil do not have the same composition, properties, and appearance throughout the mixture. Such mixtures are called *heterogeneous*. Mixtures that are uniform in composition, properties, and appearance throughout are called *homogeneous*. Such mixtures include sugar water and air. Homogeneous mixtures are also called solutions. Mixtures are characterized by two fundamental properties:

- Each of the substances in the mixture retains its chemical identity.
- Mixtures are separable into these components by physical means.

If one of the substances in a mixture is preponderant—that is, if its amount far exceeds the amounts of the other substances in the mixture—you usually call this mixture an impure substance and speak of the other substances in the mixture as impurities.

The preparation of compounds usually involves their separation or isolation from reactants or other impurities. Thus, the separation of mixtures into their components and the purification of impure substances are common problems. You are probably aware of everyday problems of this sort. For example, drinking water usually begins as a mixture of silt, sand, dissolved salts, and water. Because water is by far the largest component in this mixture, it is usually called impure

water. How is it purified? The separation of the components of mixtures is based upon the fact that each component has different physical properties. The components of mixtures are always pure substances, either compounds or elements, and each pure substance possesses a unique set of properties. The properties of every sample of a pure substance are identical under the same conditions of temperature and pressure. This means that once you have determined that a sample of sodium chloride (table salt), NaCl, is water-soluble and a sample of silicon dioxide (sand), SiO_2, is not, you realize that all samples of sodium chloride are water-soluble and all samples of silicon dioxide are not.

Likewise, every crystal of a pure substance melts at a specific temperature and a given pressure, and every pure substance boils at a specific temperature and a given pressure.

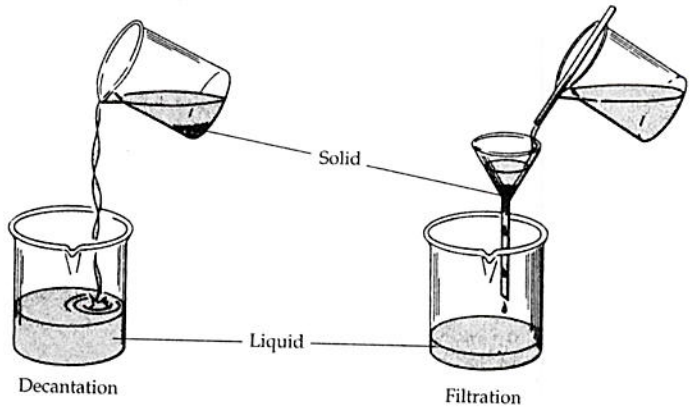
Although numerous physical properties can be used to identify a particular substance, you will be concerned in this experiment merely with the separation of the components and not with their identification. The methods you will use for the separation depend upon differences in physical properties, and they include the following:

1. *Decantation.* This is the process of separating a liquid from a solid (sediment) by gently pouring the liquid from the solid so as not to disturb the solid (Figure 3.1).
2. *Filtration.* This is the process of separating a solid from a liquid by means of a porous substance—a filter—which allows the liquid but not the solid to pass through (see Figure 3.1). Common filter materials are paper, layers of charcoal, and sand. Silt and sand can be removed from drinking water by this process.

GIVE IT SOME THOUGHT

a. When would it be best to use decantation over filtration?
b. When would you want to use filtration rather than decantation?

3. *Extraction.* This is the separation of a substance from a mixture by preferentially dissolving that substance in a suitable solvent. By this process, a soluble compound is usually separated from an insoluble compound.



▲ **FIGURE 3.1**

4. *Sublimation*. This is the process in which a solid passes directly to the gaseous state and back to the solid state without the appearance of the liquid state. Not all substances possess the ability to be sublimed. Iodine, naphthalene, and ammonium chloride (NH_4Cl) are common substances that easily sublime.

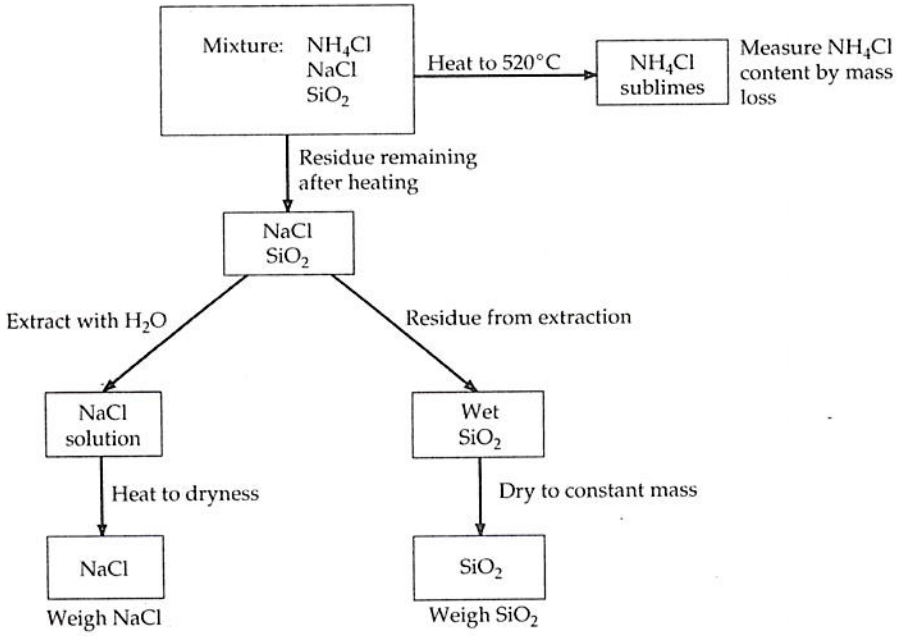
PROCEDURE

The mixture you will separate contains three components: NaCl, NH_4Cl, and SiO_2. Their separation will be accomplished by heating the mixture to sub-lime the NH_4Cl, extracting the NaCl with water, and drying the remaining SiO_2, as illustrated in the scheme shown in Figure 3.2. Carefully weigh a clean, dry evaporating dish to the nearest 0.01 g. Then obtain from your instructor a 2 to 3 g sample of the unknown mixture in the evaporating dish. Write the unknown number on your report sheet. If you obtain your unknown from a bottle, shake the bottle to make the sample mixture as uniform as possible. Weigh the evaporating dish containing the sample and calculate the sample mass to the nearest 0.001 g.

Place the evaporating dish containing the mixture on a clay triangle (or wire gauze), ring, and ring-stand assembly *In the hood* as shown in Figure 3.3. Heat the evaporating dish with a burner until white fumes no longer form (a total of about 15 min). Heat carefully to avoid spattering, especially when liquid is present. Occasionally shake the evaporating dish gently, using crucible tongs during the sublimation process.

Allow the evaporating dish to cool until it reaches room temperature; then weigh the evaporating dish with the contained solid. NEVER WEIGH HOT OR WARM OBJECTS! The loss in mass represents the amount of NH_4Cl in your mixture. Calculate this.

Add 25 mL of water to the solid in this evaporating dish and stir gently for 5 min. Then, weigh another clean, dry evaporating dish and watch glass. Decant



▲ **FIGURE 3.2** Flow diagram for the separation of the components of a mixture.

GIVE IT SOME THOUGHT

a. What property allows you to separate NH_4Cl from NaCl and SiO_2?

b. Is this property a chemical or physical property?

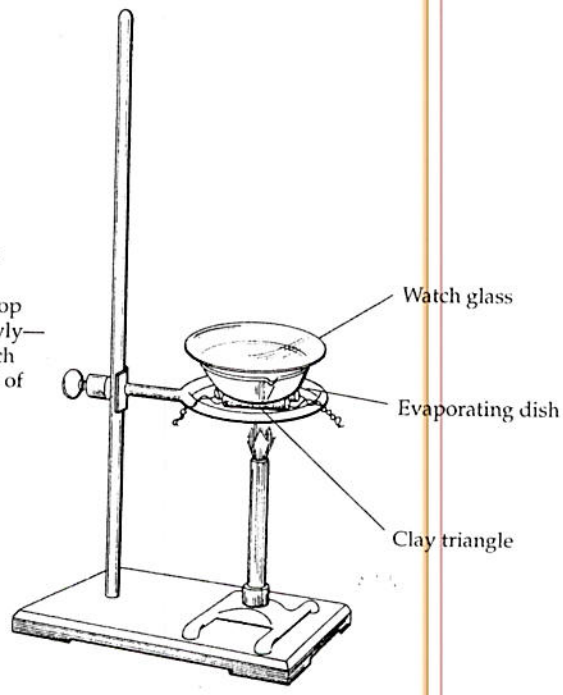
c. What property allows you to separate NaCl from SiO_2?

d. Is this a chemical or physical property?

the liquid carefully into the second evaporating dish, *which you have weighed,* being careful not to transfer any of the solid into the second evaporating dish. Add 10 mL more of water to the solid in the first evaporating dish, stir, and decant this liquid into the second evaporating dish as before. Repeat with still another 10 mL of water. This process extracts the soluble NaCl from the sand. You now have two evaporating dishes—one containing wet sand, and the other containing a solution of sodium chloride.

Carefully place the evaporating dish containing the sodium chloride solution on the clay triangle on the ring stand. Begin heating the solution gently to evaporate the water. Take care to avoid boiling or spattering, especially when liquid is present. Near the end of the process, cover the evaporating dish with the watch glass that was weighed with this evaporating dish and reduce the heat to prevent spattering. While the water is evaporating, if you have another Bunsen burner available, you may proceed to dry the SiO_2 in the other evaporating dish as explained in the next paragraph. When you have dried the sodium chloride completely, no more water will condense on the watch glass and it too will be dry. Let the evaporating dish and watch glass cool to room temperature on a wire gauze and weigh them. The difference between this mass and the mass of the empty evaporating dish and watch glass is the mass of the NaCl. Calculate this mass.

NOTE: To sublime NH_4Cl, do not use the watch glass; to dry NaCl and SiO_2, use the watch glass on top of the evaporating dish. Heat slowly—do not flame the edges of the watch glass that extend beyond the edge of the evaporating dish.



▲ **FIGURE 3.3**

GIVE IT SOME THOUGHT

How will the sum of the percentage of each substance in the mixture be influenced if you do not dry your sample properly?

Place the evaporating dish containing the wet sand on the clay triangle on the ring stand and cover the evaporating dish with a clean, dry watch glass. Heat slowly at first until the lumps break up and the sand appears dry. Then heat the evaporating dish to dull redness, maintaining this heat for 10 min. Take care not to overheat the dish; otherwise, it will crack. When the sand is dry, remove the heat and let the dish cool to room temperature. Weigh the dish after it has cooled to room temperature. The difference between this mass and the mass of the empty dish is the mass of the sand. Calculate this mass. Dispose of the sand in the marked container.

Calculate the percentage of each substance in the mixture using an approach similar to that shown in Example 3.1.

The accuracy of this experiment is such that the combined total of your three components should be approximately 99%. If it is less than 99%, you have been sloppy. If it is more than 100%, you have not sufficiently dried the sand and salt.

EXAMPLE 3.1

What is the percentage of SiO_2 in a 2.56 g sample mixture if 1.25 g of SiO_2 has been recovered?

SOLUTION: The percentage of each substance in such a mixture can be calculated as follows:

$$\%\,\text{component} = \frac{\text{mass component in grams} \times 100\%}{\text{mass sample in grams}}$$

Therefore, the percentage of SiO_2 in this particular sample mixture is as follows:

$$\%\,SiO_2 = \frac{1.25\text{ g} \times 100\%}{2.56\text{ g}} = 48.8\%$$

Name _____ Desk _____

Date _____ Laboratory Instructor _____

Separation of the Components of a Mixture | 3 | Pre-lab Questions

Before beginning this experiment in the laboratory, you should be able to answer the following questions.

1. Classify each of the following as a pure substance or a mixture; if it is a mixture, state whether it is heterogeneous or homogeneous: (a) concrete, (b) tomato juice, (c) marble, (d) seawater, and (e) iron.

2. Suggest a way to determine whether a colorless liquid is pure water or a salt solution without tasting it.

3. What distinguishes a mixture from an impure substance?

4. Define the process of sublimation.

5. How do decantation and filtration differ? Which should be faster?

6. Why should you never weigh a hot object?

7. How does this experiment illustrate the principle of conservation of matter?

8. A mixture was found to contain 1.05 g of SiO_2, 0.69 g of cellulose, and 2.17 g of calcium carbonate. What percentage of calcium carbonate is in the mixture?

9. How could you separate a mixture of acetone and α-naphthol? Consult Table 2.1 for physical properties.

10. How could you separate zinc chloride from SiO_2?

11. A student found that her mixture was 13% NH_4Cl, 18% NaCl, and 75% SiO_2. Assuming that her calculations are correct, what did she most likely do incorrectly in her experiment?

12. Why is the NaCl extracted with water three times as opposed to only once?

Name _____ Desk _____

Date _____ Laboratory Instructor _____

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REPORT SHEET | EXPERIMENT

Separation of the Components of a Mixture | 3

A. Mass of Evaporating Dish and Original Sample _____ g

Mass of evaporating dish _____ g

Mass of original sample _____ g

Mass of evaporating dish after subliming NH_4Cl _____ g

Mass of NH_4Cl _____ g

Percent of NH_4Cl (show calculations) _____ %

B. Mass of Evaporating Dish, Watch Glass, and NaCl _____ g

Mass of evaporating dish and watch glass _____ g

Mass of NaCl _____ g

Percent of NaCl (show calculations) _____ %

C. Mass of Evaporating Dish and SiO_2 _____ g

Mass of evaporating dish _____ g

Mass of SiO_2 _____ g

Percent of SiO_2 (show calculations) _____ %

D. Mass of Original Sample

Mass of determined $(NH_4Cl + NaCl + SiO_2)$ _____ g

Differences in these weights _____ g

Percent recovery of matter $= \frac{\text{g matter recovered}}{\text{g original sample}} \times 100\% =$ _____ g

_____ %

Account for your errors.

QUESTIONS

1. Could the separation in this experiment have been done in a different order? For example, if the mixture was first extracted with water and then both the extract and the insoluble residue were heated to dryness, could you determine the amounts of NaCl, NH_4Cl, and SiO_2 originally present? Why or why not?

Consult a handbook to answer these questions.

2. How could you separate barium sulfate, $BaSO_4$, from NaCl?

3. How could you separate magnesium chloride, $MgCl_2$, from silver chloride, AgCl?

4. How could you separate tellurium dioxide, TeO_2, from SiO_2?

5. How could you separate lauric acid from α-naphthol? (See Table 2.1.)