Separation of the Components of a Mixture

EXPERIMENT

3

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)
iron rings (2)
glass stirring rods

Chemicals

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

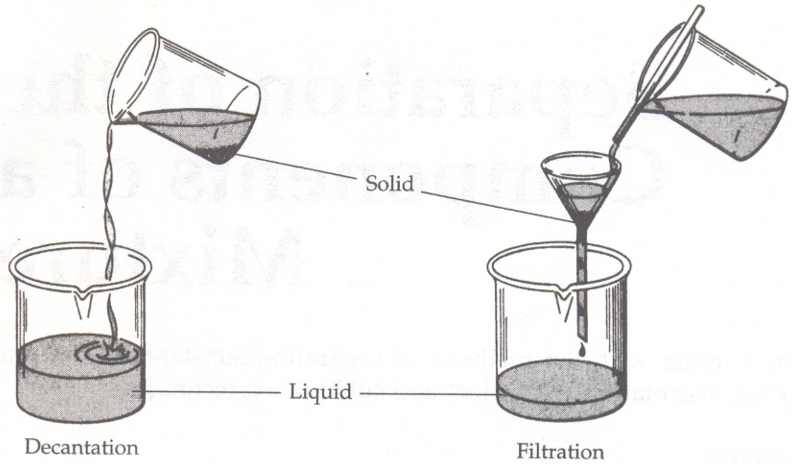
DISCUSSION

Materials that are not uniform in composition are said to be impure or heterogeneous and are called *mixtures.* Most of the materials we encounter in everyday life, such as cement, wood, and soil, are mixtures. When two or more substances that do not react chemically are combined, a mixture results. Mixtures are characterized by two fundamental properties:

- Each of the substances in the mixture retains its chemical integrity
- 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—then we 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 frequently encountered problems. You are probably aware of everyday problems of this sort. For example, our drinking water usually begins as a mixture of silt, sand, dissolved salts, and water. Since water is by far the largest component in this mixture, we usually call this impure water. How do we purify it? 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 we have determined that a



▲ **FIGURE 3.1**

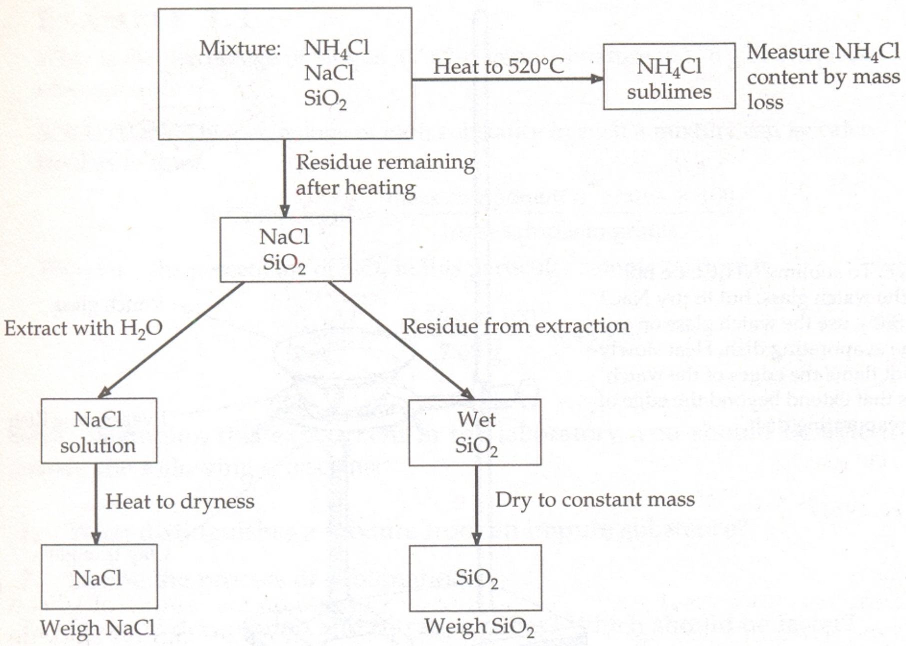
sample of sodium chloride, NaCl, is water-soluble and a sample of silicon dioxide (sand), SiO_2, is not, we realize that all samples of sodium chloride are water-soluble and all samples of silicon dioxide are not.

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

Although there are numerous physical properties that can be used to identify a particular substance, we will be concerned in this experiment merely with the separation of the components and not with their identification. The methods we will use for the separation depend on 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 to pass through but not the solid (see Figure 3.1). Common filter materials are paper, layers of charcoal, and sand. Silt and sand can be removed from our drinking water by this process.
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.
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.

The mixture that you will separate contains three components: NaCl, NH_4Cl, and SiO_2. Their separation will be accomplished by heating the mixture to sublime the NH_4Cl, extracting the NaCl with water, and finally drying the remaining SiO_2, as illustrated in the scheme shown in Figure 3.2.



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

PROCEDURE

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. 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.

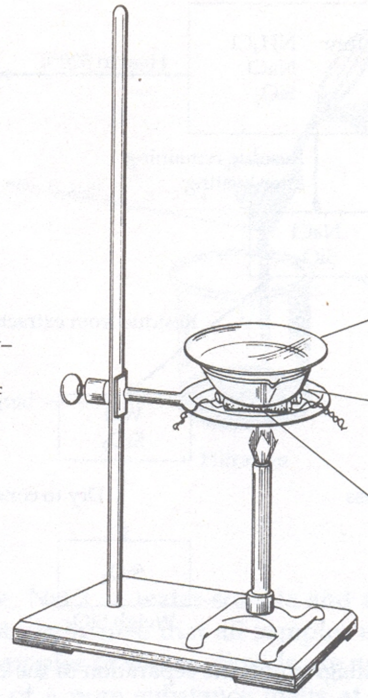
Place the evaporating dish containing the mixture on a clay triangle (or wire gauze), ring, and ring-stand assembly IN THE HOOD as shown in Figure 3.3. Heat the evaporating dish with a burner until white fumes are no longer formed (a total of about 15 min). Heat carefully to avoid spattering, especially 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 and 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. Next, weigh another clean, dry evaporating dish and watch glass. Decant 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 second, a solution of sodium chloride.

Place the evaporating dish containing the sodium chloride solution carefully on the clay triangle on the ring stand. Begin gently heating the solution to evaporate the water. Take care to avoid boiling or spattering, especially

NOTE: To sublime NH_4Cl, do not use the watch glass; but 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

when liquid is present. Near the end, 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 you may proceed to dry the SiO_2 in the other evaporating dish as explained in the next paragraph, if you have another Bunsen burner available. 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 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.

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 and maintain this heat for 10 min. Take care not to overheat, or the evaporating dish 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 in the neighborhood of 99%. If it is less than this, 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 7.69-g sample mixture if 3.76 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

$$\% \ SiO_2 = \frac{3.76 \text{ g} \times 100}{7.69 \text{ g}} = 48.9\%$$

REVIEW QUESTIONS

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

1. What distinguishes a mixture from an impure substance?
2. Define the process of sublimation.
3. How do decantation and filtration differ? Which should be faster?
4. Why does one never weigh a hot object?
5. How does this experiment illustrate the principle of conservation of matter?
6. A mixture was found to contain 3.10 g of SiO_2, 0.38 g of cellulose, and 6.72 g of calcium carbonate. What is the percentage of SiO_2 in this mixture?
7. How could you separate a mixture of zinc chloride and cyclohexane? Consult Table 2.1 for physical properties.
8. How could you separate zinc chloride from SiO_2?
9. A student found that her mixture was 15% NH_4Cl, 20% NaCl, and 75% SiO_2. Assuming her calculations are correct, what did she most likely do incorrectly in her experiment?
10. Why is the NaCl extracted with water three times as opposed to only once?

NOTES AND CALCULATIONS

Name ______________________________ Desk ____________

Date ____________________ Laboratory Instructor ____________________

Unknown no. ____________

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 ______________ g

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

Differences in these weights ______________ g

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

Account for your errors

QUESTIONS

1. Could the separation in this experiment have been done in a different order? For example, if the mixture were first extracted with water and then the extract and the insoluble residue both 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 NH_4Cl?

3. How could you separate zinc chloride, $ZnCl_2$, from zinc sulfide, ZnS?

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

5. Naphthalene sublimes easily but potassium bromide does not. How could you separate these two substances?