

Separation of the Components of a Mixture

EXPERIMENT

3

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

OBJECTIVE

balance
Bunsen burner and hose
clay triangles (2)
evaporating dishes (2)
glass stirring rod
50- or 100-mL graduated cylinder
iron rings (2)

ring stands (2)
tongs
watch glass
unknown mixture of sodium
chloride, ammonium chloride,
and silicon dioxide (2–3g)

APPARATUS AND CHEMICALS

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: First, each of the substances in the mixture retains its chemical integrity; second, 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.

DISCUSSION

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 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 (as you learned in the previous experiment), 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 upon differences in physical properties, and they include the following:

1. *Decantation*. This is the process of separation of a liquid from a solid (sediment) by gently pouring the liquid from the solid so as not to disturb the solid (see 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.

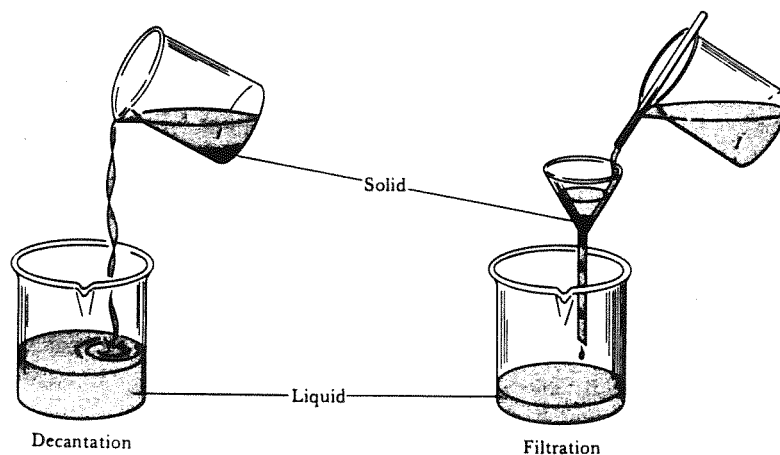


Figure 3.1

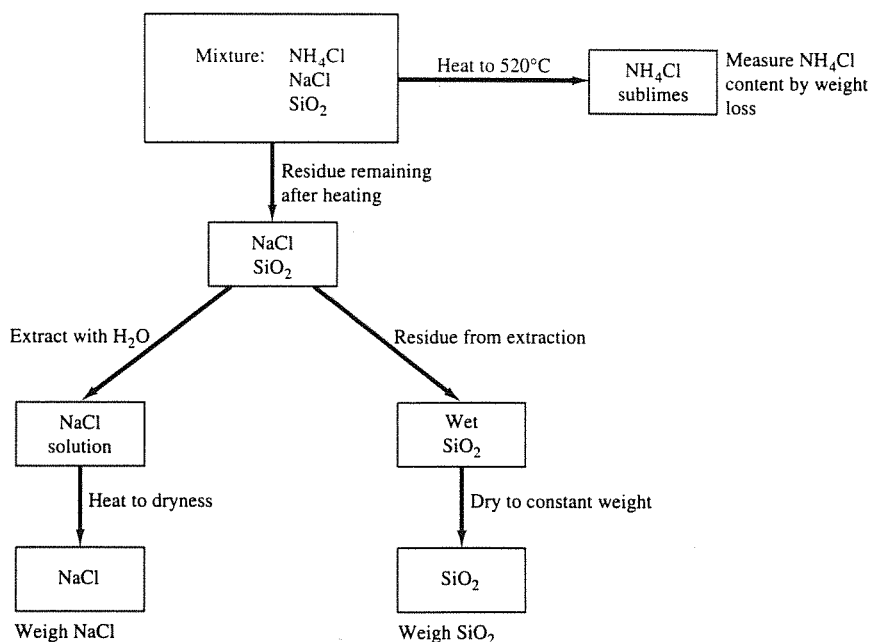


Figure 3.2 Flow diagram for the separation of the components of a mixture.

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. Weigh the evaporating dish containing the sample and calculate the sample weight.

PROCEDURE

Place the evaporating dish containing the mixture on a clay triangle, 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

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.

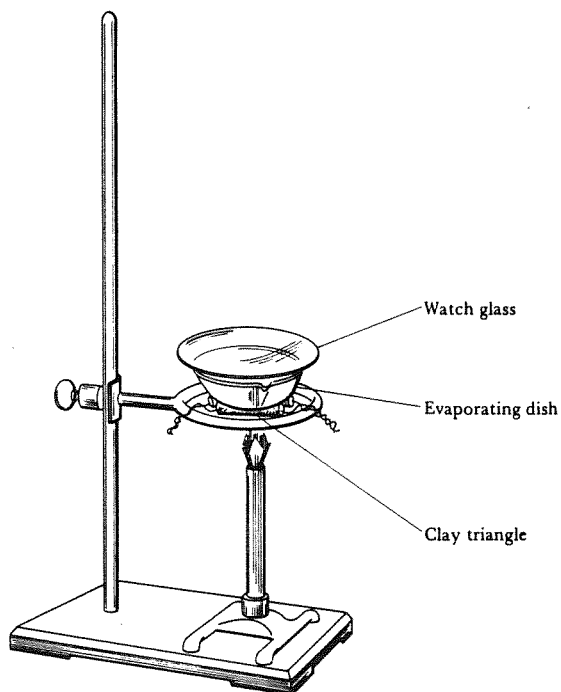


Figure 3.3

of about 15 min). Heat carefully to avoid spattering, especially when liquid is present. After the first 10 min remove the flame and gently stir the mixture with your glass stirring rod; then apply the heat again.

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 weight 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 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 weight and the weight of the empty evaporating dish and watch glass is the weight of the NaCl . Calculate this weight.

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 weight and the weight of the empty dish is the weight of the sand. Calculate this weight.

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 percent. If it is less than this, you have been sloppy. If it is more than 100 percent, 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:

$$\% \text{ Component} = \frac{\text{wt component in grams} \times 100}{\text{wt sample in grams}}$$

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

$$\% \text{ SiO}_2 = \frac{3.76 \text{ g} \times 100}{7.69 \text{ g}} = 48.9\%$$

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 2.10 g of SiO_2 , 0.38 g of cellulose, and 7.52 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 percent NH_4Cl , 20 percent NaCl , and 75 percent SiO_2 . Assuming her calculations are correct, what did she most likely do incorrectly in her experiment?
10. Why is the SiO_2 extracted with water three times as opposed to only once?

Name _____ Desk _____

Date _____ Laboratory Instructor _____

Unknown no. _____

REPORT SHEET FOR EXPERIMENT 3

SEPARATION OF THE COMPONENTS OF A MIXTURE

A. Weight of evaporating dish and original sample _____ g
Weight of evaporating dish _____ g
Weight of original sample _____ g
Weight of evaporating dish after subliming NH_4Cl _____ g
Weight of NH_4Cl _____ g
Percent of NH_4Cl (show calculations) _____ %

B. Weight of evaporating dish, watch glass, and NaCl _____ g
Weight of evaporating dish and watch glass _____ g
Weight NaCl _____ g
Percent of NaCl (show calculations) _____ %

C. Weight of evaporating dish and SiO_2 _____ g
Weight of evaporating dish _____ g
Weight of SiO_2 _____ g
Percent of SiO_2 (show calculations) _____ %

D. Weight of original sample _____ g
 Weight of determined (NH₄Cl + NaCl + SiO₂) _____ 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, NH₄Cl, and SiO₂ originally present? Why or why not?

Consult a handbook to answer these questions.

2. How could you separate barium sulfate, BaSO₄, from NH₄Cl?
3. How could you separate zinc chloride, ZnCl₂, from zinc sulfide, ZnS?
4. How could you separate tellurium dioxide, TeO₂, from SiO₂?
5. Naphthalene sublimes easily but potassium bromide does not. How could you separate these two substances?

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Percent of NH_4Cl (show calculations) _____ %

B. Weight of evaporating dish, watch glass, and NaCl _____ g
Weight of evaporating dish and watch glass _____ g
Weight NaCl _____ g
Percent of NaCl (show calculations) _____ %

C. Weight of evaporating dish and SiO_2 _____ g
Weight of evaporating dish _____ g
Weight of SiO_2 _____ g
Percent of SiO_2 (show calculations) _____ %

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