Separation of Components of a Mixture

Objective

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

Equipment

Balance

Clay triangle or wire gauze

Watch glass

Evaporating dishes (2)

Crucible tongs

Bunsen burner and hose

Striker

Ring stand

Iron ring

DI water

50- or 100-mL graduated cylinder

Glass stirring rod

Chemicals

Unknown mixture of sodium chloride (NaCl), ammonium chloride (NH_4Cl), and silicon dioxide (SiO_2)

Introduction

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. For example, a glass of sweetened tea may contain 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***. *Homogeneous* mixtures are also called solutions. Such mixtures include sugar-water solution, and air. 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 in excess, it is an impure substance and containing impurities (i.e. the minor components).

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 substances, either compounds or elements, and each substance possesses a unique set of properties. The properties of every sample of a 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 can use for the separation depend upon differences in physical properties, and 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 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 1**). Common filter materials are paper, layers of charcoal, and sand. Silt and sand can be removed from 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 statewithout 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.

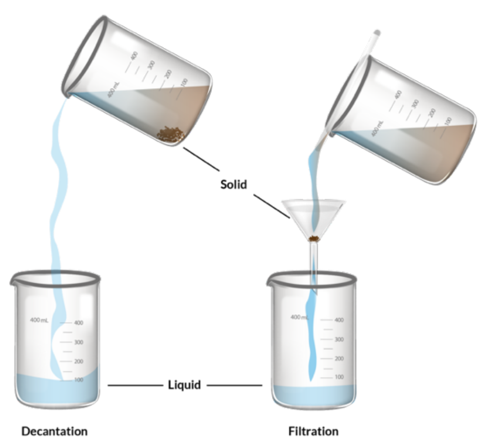

Figure 1 Difference between decantation and filtration

Procedure

You will be working in pairs for this experiment. The mixture 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 drying the remaining SiO_2, as illustrated in **Figure 2**.

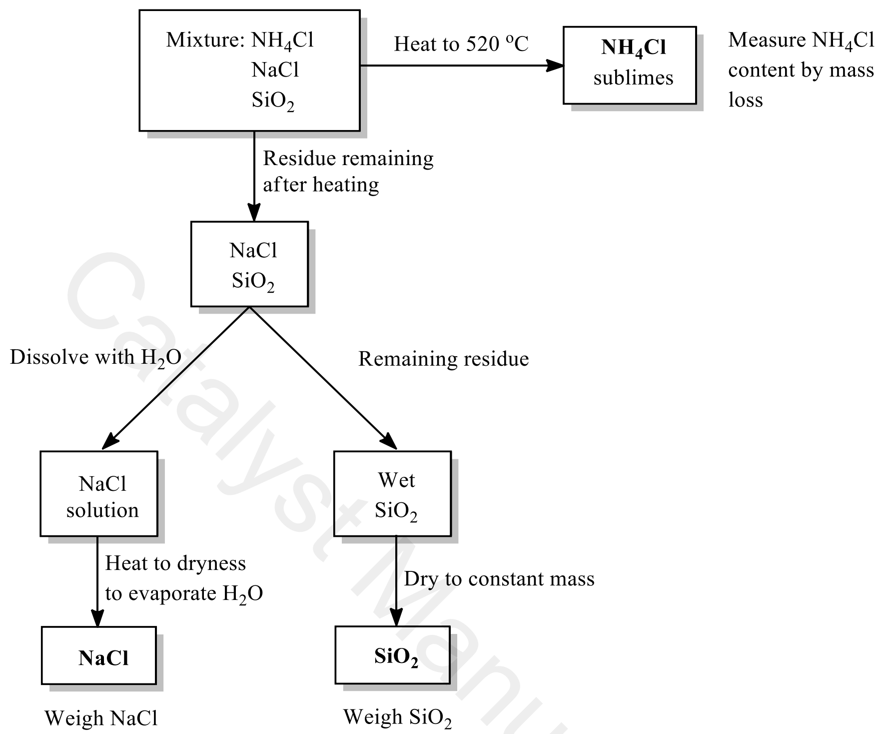

Figure 2 Flow diagram for the separation of the components of a mixture.

1. Carefully weigh a dry and clean evaporating dish #1 to the nearest 0.001 g. Record this measurement in your report sheet. **[1]** Then obtain from your instructor a sample of the unknown mixture. **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 ~ 0.8 to 1.0 g of the sample in the evaporating dish and record the sample mass to the nearest 0.001 g. **[2]**
2. In the fume hood, place the evaporating dish containing the mixture on a clay triangle (or wire gauze), ring, and ring-stand assembly as shown in **Figure 3**. Heat the evaporating dish with a Bunsen burner until white fumes no longer form (about 15 min). Heat carefully to avoid spattering. Occasionally, gently shake the evaporating dish, using crucible tongs during the sublimation process to ensure sublimation is complete.
3. Allow the evaporating dish to cool until it reaches room temperature; then weigh the evaporating dish with the contained solid. **[3]** **NEVER WEIGH HOT OR WARM OBJECTS** The loss in mass represents the amount of NH_4Cl in your mixture. Calculate this.

4. Weigh another dry and clean evaporating dish #2 **[4]** and watch glass **[5]** as lid. Remove the salt from the sand by adding small portions of DI water to the sand-salt mixture (evaporating dish #1). With a stirring rod gently, stir the mixture. Decant the liquid carefully into the evaporating dish #2, *which you have weighed*, being careful not to transfer any of the solid into evaporating dish #2.

5. Add more DI water to the solid in evaporating dish #1, stir, and decant this liquid into evaporating dish #2 as before. Repeat the process a total of three times. Use no more than 25 mL of DI water. This process dissolves (extracts) the NaCl from the sand. You now have two evaporating dishes - one containing wet sand, #1, and the other containing a solution of sodium chloride, #2.

6. Carefully place evaporating dish #2, which contains 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 evaporating dish #2 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 evaporating dish #1 as explained in step VIII. When you have dried the sodium chloride completely, no more water will condense on the watch glass and it too will be dry.

7. Let the evaporating dish and watch glass cool to room temperature on a wire gauze and weigh them. **[6]** 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.

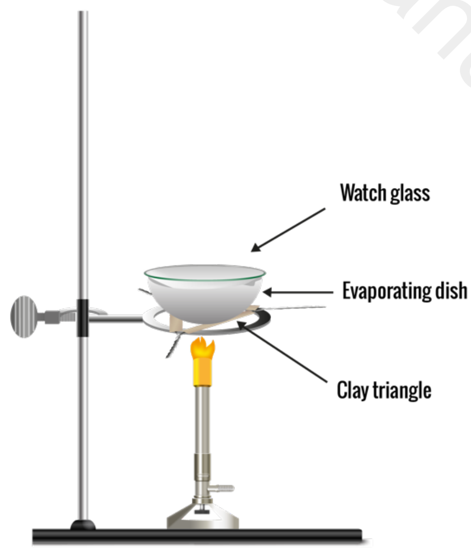

Figure 3 Correct assembly of the evaporating dish on the ring stand

> ***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.*

8.Place evaporating dish #1, containing the wet sand, on a clay triangle on a ring stand and cover the evaporating dish with a clean, dry watch glass. **[7]** Heat slowly at first until the lumps break up and the sand appears dry. Then heat evaporating dish #1 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 evaporating dish #1 cool to room temperature. Weigh evaporating dish #1 after it has cooled to room temperature. **[8]** 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.

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

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

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:

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

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

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

Name: ____ Section: _________

Laboratory Instructor: ____________________________________ Date: ___/___/___

Report Sheet: Separation of Components of a Mixture

Unknown number

A) Determination of NH_4Cl

[1] Mass of evaporating dish #1 ___________

[2] Mass of evaporating dish and original sample ___________

Mass of original sample ___________

[3] Mass of evaporating dish after subliming NH_4Cl ___________

Mass of NH_4Cl ___________

Percent of NH_4Cl (show calculations) ___________

Show calculation of percent NH_4Cl:

B. Determination of NaCl

[4] Mass of evaporating dish #2 ___________

[5] Mass of watch glass ___________

[6] Mass of evaporating dish #2, watch glass, and NaCl ___________

Mass of NaCl

Percent of NaCl (show calculations) ____________

Show calculation of percent NaCl:

C. Determination of SiO_2

[1] Mass of evaporating dish #1 ____________

[7] Mass of watch glass ____________

Mass of SiO_2 ____________

Percent of SiO_2 (show calculations) ____________

Show calculation of percent SiO_2:

Mass of Original Sample ____________

Experimental mass recovered of (NH_4Cl + NaCl + SiO_2) ____________

Differences in these weights ____________

$$\% \, recovery \ of \ matter = \frac{g \ matter \ recovered}{g \ original \ sample} x \ 100\%$$ ____________

Show calculations

Account for your errors: