EXPERIMENT #3
Separation of a Three-Component Mixture

OBJECTIVES:
- Define chemical and physical properties, mixture, solubility, filtration, sublimation, and percent
- Separate a mixture of sodium chloride (NaCl), ammonium chloride (NH₄Cl), and sand (SiO₂) by sublimation and filtration
- Report the percent of each component in the mixture.

BACKGROUND:
Chemistry is the science dealing mostly with the changes of one substance into another -- changes in composition. Properties associated with the change of one substance into another are called chemical properties. Chemistry is also associated with properties such as color, state of subdivision, density, crystal structures of solids, and conditions determining whether a substance is in the form of a solid, liquid, or gas. These properties, and others not related to a change in composition, are called physical properties. Sodium is a reactive metal and forms sodium hydroxide and hydrogen gas upon reaction with water. The reaction of sodium with water is a chemical property. On the other hand, sodium is a soft metal, which is readily sliced with a knife, and possesses a relatively low melting point. The melting point and consistency of sodium are physical properties, because they are not associated with the conversion of sodium into another substance.

Most of the materials we see around us are mixtures. A mixture is composed of two or more substances which can be separated by physical methods. This means we can separate the components of a mixture without converting one substance into another. Methods of separating mixtures are limited only by the imagination of the chemist and the physical properties of each component.

In this experiment the components of the mixture are known. The mixture consists of three substances: sodium chloride, NaCl; ammonium chloride, NH₄Cl; and sand, SiO₂. Let's look at a brief list of physical properties of these materials.

<table>
<thead>
<tr>
<th></th>
<th>NaCl</th>
<th>NH₄Cl</th>
<th>SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>solubility, g/100 g H₂O at 25°C</td>
<td>35</td>
<td>37</td>
<td>insoluble</td>
</tr>
<tr>
<td>melting point, °C</td>
<td>801</td>
<td>350</td>
<td>1600</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>sublimes</td>
</tr>
</tbody>
</table>

Solubility is a physical property designating the number of grams of substance which will dissolve in 100 g of water at a certain temperature, in this case 25°C. Melting point is a physical property describing the temperature at which a solid is in equilibrium with its liquid. Sublimation is a physical process in which a solid is converted to a gas without going through the liquid state. Solid carbon dioxide (dry ice), iodine, and menthol are some common substances subliming at atmospheric pressure and room temperature.

While there are several strategies for separating these components, we will use a method which can be completed in a two hour laboratory period. By heating the mixture strongly, but below the melting point of sodium chloride and sand, ammonium chloride is removed by sublimation. The ammonium chloride is converted to the gaseous phase, and the gas is removed in the exhaust of the fume hood (FIGURE I). The loss in mass is equal to the mass of ammonium chloride originally
The two remaining components can be separated based on their solubility – sodium chloride will dissolve in water while sand will not. The sand can be separated from the aqueous solution of sodium chloride by the technique of filtration. Filtration is used to separate a solid from a liquid. The liquid passes through a porous barrier, such as a piece of filter paper, and the solid remains behind. In our case, the aqueous solution will pass through the filter paper (FIGURE II), while the sand will remain behind. The sand will be dried and weighed affording the mass of a second component. Subtracting the mass of the ammonium chloride and the mass of the sand from the mass of the original mixture gives the mass of the sodium chloride.

Once we know the mass of each component, we can report its percentage. Percent is a common measurement, and its mathematical form is always the same:

\[
\text{percent} = \frac{\text{"part"}}{\text{"whole"}} \times 100
\]

Each percentage problem differs only by what is defined as the "whole" and what is defined as the "part." In this experiment the "whole" is the mass of the original mixture, while the "part" is the mass of each component. Since the "whole" is equal to the sum of its "parts," all three percentages must add up to 100%, if the experiment and calculation have been performed correctly.

**Example:**

What is the percentage of sand in a 7.31 g sample mixture, if 3.59 g of sand has been recovered. What is the percentage of the other components in the mixture?

\[
\text{percentage of sand} = \frac{\text{mass of sand recovered}}{\text{mass of sample mixture}} \times 100
\]

\[
\text{percentage of sand} = \frac{3.59 \text{ g}}{7.31 \text{ g}} \times 100
\]

% sand = 49.1%

percentage of other components = 100.0% - % sand

= 100.0% - 49.1% = 50.9%
**PROCEDURE:** (Work in pairs.)

1. Weigh an evaporating dish to ±0.001 g. Record the mass on your data sheet (line 3).

2. Weigh approximately 2 grams of assigned sample, to the same precision as above. Record the sample letter on the Data Sheet. Weigh the dish and sample to the nearest thousandth of a gram. Record the mass of the dish and sample on the data sheet (line 1).

3. Place the dish on a clay triangle (in the hood) and cover with a watch glass (**FIGURE I**). Heat gently at first, then strongly for at least 10 minutes. The sample may glow red. Don't try to take a shortcut at this point. You can heat too little, but not too much. The ammonium chloride sublimes from the sample. (The white solid, which forms on the watch glass, will disappear, if the strong heating is performed properly.) A small error here can result in a large error in the percentage calculation later.

4. Allow dish to cool to room temperature. Reweigh dish and sample. Record the mass on your data sheet (line 2).

5. Weigh a piece of filter paper to the nearest thousandth of a gram. Record mass (line 7). Fold the filter paper into quarters and place it in a funnel so that one side of the funnel has three folds of paper and the other side of the funnel has one fold of paper. Wet the paper with deionized water in order to hold it in place in the funnel.

6. Add 10 - 15 mL of deionized water to the dish and stir for five (5) minutes. Using the stirring rod to direct the slurry, pour the mixture into the filter always keeping the liquid level below the top edge of the filter paper. Repeat this step two more times.

7. Rinse sand from dish into filter by directing a stream of distilled water form a wash bottle into the dish.

8. Carefully transfer filter paper with sand to a clean watch glass and dry in oven.

9. When dry and cool, weigh paper and sand. Record mass (line 6).

10. Complete lines 4, 5, 8, and 9. Calculate percentage of each component.

**11. SHOW EACH OF YOUR CALCULATIONS CLEARLY.**
**FLOW CHART:**

NaCl, SiO₂, NH₄Cl
--determine mass of mixture
--heat strongly

NaCl, SiO₂
--treat with water
--filter

NH₄Cl
--determine mass of sublimate

NaCl solution
--determine mass of NaCl by difference

SiO₂
--collect on filter
--dry
--determine mass of sand

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**FIGURE I:** Set-Up for Removing NH₄Cl

**FIGURE II:** Filtration Set-Up
DATA AND CALCULATIONS: Mixture

Letter/Number of unknown sample __________

1. Mass of evaporating dish and sample before heating ________________

2. Mass of evaporating dish and sample after heating ________________

3. Mass of evaporating dish ________________

4. Mass of original sample (1 - 3) ________________

5. Mass of NH₄Cl (1 - 2) ________________

6. Mass of filter paper and sand ________________

7. Mass of filter paper ________________

8. Mass of sand (6 - 7) ________________

9. Mass of salt [4 - (5 + 8)] ________________

**CLEARLY** show your calculations of the percentage of each component in your sample below.

%NH₄Cl__________________  %Sand__________________  %NaCl__________________
ADDITIONAL ASSIGNMENT I: Mixture

1. What is the percentage of sand in a 7.69 g sample mixture if 3.76 g of sand have been recovered?

2. A mixture was found to contain 2.123 g of sand, 0.287 g of cellulose, and 7.861 g of calcium carbonate. What is the percentage of calcium carbonate in this mixture?

3. In the ethyl alcohol molecule, 34.8% of the mass is due to oxygen. What is the mass of oxygen in 497 g of alcohol?
4. If a solution is 23.3% by mass HCl and it contains 14.8 g of HCl, what is the total mass of the solution?

5. The copper content of a normal healthy human person is approximately $1.1 \times 10^{-4}$ percent by mass. How many grams of copper would exist in a person weighing 150 lb. ($1.0 \text{ kg} = 2.2 \text{ lb}$)

xc. The thin outer layer of Earth, called the crust, contains only 0.50 percent of Earth’s total mass and yet is the source of almost all the elements (the atmosphere provides elements such as oxygen, nitrogen, and a few other gases). Silicon (Si) is the second most abundant element in Earth’s crust (27.2 percent by mass). Calculate the mass of silicon in kilograms in Earth’s crust. (The mass of Earth is $5.9 \times 10^{21}$ tons. 1 ton = 2000 lb; 1 lb = 453.6 g.)