EXPERIMENT 2 – Separation of a Mixture

INTRODUCTION

The physical separation of the components of a mixture is the goal of this experiment. Each component in a mixture retains its individual physical properties. That is, they do not react with each other (a chemical change) in the mixture or during the separation process. Product mixtures are a common result in chemical reactions. Indeed, it is rare that a reaction results in a single, pure product directly. The real challenge often is the separation of the desired product from the mixture and its subsequent purification.

You will rely on differences in properties to separate silicon dioxide (SiO_2, sand), sodium chloride (NaCl, table salt), and ammonium chloride (NH_4Cl), from a mixture of these solid compounds. Three commonly-used methods of separation are given below:

(a) SUBLIMATION

Sublimation is the process by which a solid changes from the solid to the gaseous state directly without forming a liquid. *Melting* is a process by which a solid changes to a liquid by heating. In the mixture used in this experiment, one compound, ammonium chloride sublimes easily, while the other two components do not.

(b) SOLUBILITY

The extent to which a substance is soluble in a solvent depends upon the chemical structure of both the substance and the solvent. In general, polar compounds, such as sugar and alcohol, and ionic compounds, such as KCl, NaCl, NH_4Cl, and NH_4NO_3, are soluble in polar solvents such as water. Nonpolar substances such as grease, wax, and oil, are soluble in nonpolar solvents such as toluene or kerosene. Extracting (dissolving) a soluble substance out of a mixture with a appropriate solvent is a common separation technique. In this experiment you will use solubility to extract a solid, NaCl, which is soluble in water, from another solid, SiO_2, which is insoluble in water.

(c) DISTILLATION

If two components have very different boiling points, the substance with the lower boiling point will evaporate more rapidly at a given temperature than the substance with a higher boiling point, and so they can be separated on this basis. Solid NaCl with a very high boiling point can be separated from a solution (NaCl–H_2O solution) by simply evaporating the water, which has a much lower boiling point. The solid NaCl will remain in the dish as a dry residue. If the water vapor is condensed to a liquid and is collected, the process is called *distillation*.

The separation techniques of sublimation, extraction, and evaporation usually do not change the chemical composition of a substance, and as such, are standard separation techniques used by chemists.

Experimental Procedure

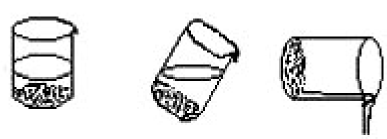
Part I
Obtain a sample of "unknown mixture" from your instructor and record its identity number or letter on the report form. Weigh a clean, dry evaporating dish to the nearest 0.01 g. Record the mass on the report form. Measure approximately 2 g of your mixture by carefully adding it directly to the evaporating dish while it is on the balance. The mass of sample does not have to be exactly two grams, but you should measure the mass to within +/– 0.01 g.

Part II
Heat the evaporating dish and its contents on an electric hot plate in the fume hood. Ammonium chloride, NH_4Cl, will sublime from the mixture and appear as a white smoke. Continue heating until the white smoke is no longer produced. At this point, you can stir the hot mixture with a glass stirring rod to ensure complete sublimation of the ammonium chloride. Allow the evaporating dish and its contents to cool to room temperature and weigh them together to the nearest 0.01 g. Record this mass on the report form. From the differences in masses before and after heating, determine the mass of NH_4Cl and calculate its percent by mass.

$$\textbf{Percent by mass of “Z”} = \frac{\textbf{mass of Z}}{\textbf{total mass of the sample}} \times 100$$

Part III
Add about 20 mL of deionized water to the residue in the evaporating dish to dissolve the NaCl. Stir the mixture well for a few minutes with a glass stirring rod to dissolve the NaCl completely. Next, *decant* the water (now a solution of dissolved NaCl) into a second, pre-weighed evaporating dish, leaving the undissolved solid (sand, SiO_2) behind in the original evaporating dish. Add about 10 mL of deionized water to the wet sand, stir well with the stirring rod as before, and decant the liquid into the second evaporating dish, which will now contains about 30 mL of water with dissolved NaCl. Set this dish and solution aside for Part IV.

Decantation:



Heat the evaporating dish containing the wet sand on the hot plate to dry the sand. Use *crucible* tongs (not *beaker* tongs!) to quickly lift the dish from the plate when needed, as the last remainder of water can evaporate suddenly and cause the sand to "popcorn" out of the dish like popping popcorn! Alternately, you can cover the dish with a watch glass (concave side downward) to prevent any sand from spattering out, but some sand may stick to the watch glass.

Allow the evaporating dish and dry sand to cool to room temperature, and if necessary, carefully scrape any sand sticking to the watch glass back into the dish. Weigh the dish and sand to the nearest 0.01 g and record this mass on the report form. Determine the mass of SiO_2 and calculate its percent by mass in the sample.

Part IV

Heat the second evaporating dish containing the salt water solution on the hot plate. You should not heat so strongly that the solution splashes out of the container when it is boiling, but it is not necessary to cover the dish with a watch glass, as this will inhibit the evaporation of the water. The goal is to evaporate the water completely, leaving a residue of dry NaCl in the dish. However, when the salt is nearly dry, it has a strong tendency to "popcorn" (see note above), causing loss of NaCl to occur. There are two ways to prevent this. You can use your crucible tongs to lift the dish off and on the hot plate to control the rate of evaporation of the last of the water, or, you can place a watch glass on the evaporating dish and let any spattering salt stick to the underside of the watch glass. Either way, after the salt appears dry and no more spattering is occurring, continue heating the open dish, and the watch glass also if it has any salt adhering to it, for about 10 minutes. This extra heating is done to ensure that the salt is completely dry. Allow the evaporating dish and dry NaCl, and the watch glass if it was used, to cool to room temperature. Carefully scrape any salt adhering to the dry watch glass back into the dish. Weigh the dish and salt to the nearest 0.01 g and record this mass on the report form. Calculate the mass of NaCl and its percent by mass in the sample.

Part V

Add up the masses of NH_4Cl, SiO_2, and NaCl from Parts II, III, and IV, and record this total mass on the report form. From this, calculate the total percentage of mixture recovered in this experiment.

$$\textbf{Percent Recovery} = \frac{\textbf{mass of } NH_4Cl + \textbf{mass of } SiO_2 + \textbf{mass of NaCl}}{\textbf{original mass of sample}} \times 100$$

Ideally, your percent recovery should be in the neighborhood of 100 percent. If your percentage recovery is less than 100 percent, *or more* than 100 percent, give an explanation for the error on the report sheet.

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REPORT FORM

Name ____________________________

Instructor _________________________

Date ____________________________

Part I

1) Sample Number _____

2) Mass of empty evaporating dish ________ g

3) Mass of dish + mixture ________ g

4) Mass of mixture ________ g

Part II

5) Mass of dish + sand + salt (after subliming the NH_4Cl) ________ g

6) Mass of ammonium chloride [(3) – (5)] ________ g

7) Percentage of NH_4Cl ________ %
(show your calculation)

Part III

8) Mass of dish + dry SiO_2 (after heating) ________ g

9) Mass of dry SiO_2 [(8) – (2)] ________ g

10) Percentage of SiO_2 ________ %
(show your calculation)

Part IV

11) Mass of empty <u>second</u> evaporating dish ________ g

12) Mass of evaporation dish + dry NaCl (after heating) ________ g

13) Mass of dry NaCl [(12) – (11)] ________ g

14) Percentage of NaCl ________ %
(show your calculation)

Part V

15) Total mass of NH_4Cl, SiO_2, and NaCl ________ g

16) Percent recovery ________ %
Explanation:

EXPERIMENT 2 **Name:**

Pre-Laboratory Questions and Exercises

Due before lab begins. Answer in the space provided.

1. Define the following terms;

a) Evaporation

b) Sublimation

2. A student used 10 mL water instead of 30 mL for the extraction of the NaCl from the mixture. How would this affect the calculated percentage of NaCl in the sample?

3. Naphthalene, $C_{10}H_8$, is insoluble in water but sublimes easily when heated. How could you separate a mixture of naphthalene and NaCl?

4. Define the process of *boiling* (as opposed to evaporation).

5. A mixture was found to contain 1.50 g of NH_4Cl, 0.80 g of NaCl, and 1.20 g of SiO_2. What is the percentage of sand in this mixture?

EXPERIMENT 2 **Name:**

Post-Laboratory Questions and Exercises

Due after completing the lab. Answer in the space provided.

1. Define the following terms:

a) Filtration

b) Decantation

2. During heating, some solution of NaCl splattered out of the evaporating dish. How would this affect the percent recovery of the mixture?

3. What is the difference between a *mixture* and a *pure substance*?

4. Is the *melting* temperature of a substance different from its *freezing* temperature? ________
Define the process of melting:

5. A student reported the following percentages for his unknown mixture: 25% NH_4Cl, 30% NaCl, and 50% SiO_2. Assuming that the calculations were done correctly, explain the student's error.