Lab #2: Physical Separation Techniques

INTRODUCTION

When two or more substances that do not react chemically are blended together, the result is a mixture in which each component retains its individual identity and properties.

The separation of the components of a mixture is a problem frequently encountered in chemistry. The basis of the separation is the fact that each component has a different set of physical and chemical properties. The components are pure substances that are either elements or compounds. Under the same conditions of pressure and temperature, the properties of every sample of a pure substance are identical. Each sample melts at the same temperature, boils at the same temperature, has the same solubility in a given solvent, etc.

Although these and other characteristics can be used to identify a particular substance, we will be concerned, in this experiment, with the separation of a mixture into its components, not with the identification of the substances. Techniques used to separate mixtures rely on differences in the physical properties of the components. Techniques useful for the separation of mixtures, include the following:

Distillation is the purification of a liquid by heating it to its boiling point, causing vaporization, and then condensing the vapors into the liquid state and collecting the liquid. Separation of two or more liquids requires that they have different boiling temperatures. Decreasing the pressure on the liquid can reduce all boiling temperatures.

Extraction is the removal of one substance from a mixture because of its greater solubility in a given solvent.

Filtration is the process of removing or "straining" a solid (the chemical term is precipitate) from a liquid by the use of filter paper or other porous material.

Decanting is the pouring of a liquid from a solid-liquid mixture, leaving the solid behind.

Sublimation is the physical property of some substances to pass directly from the solid state to the gaseous state without the appearance of the liquid state. Not all substances possess this characteristic. If one component of a mixture sublimates, this property may be used to separate it from the other components of the mixture. Iodine (I_2), naphthalene ($C_{10}H_8$, mothballs), ammonium chloride (NH_4Cl) and dry ice (solid CO_2) are some substances that sublime.

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Centrifuging is the process of separating a suspended solid from a liquid by whirling the mixture at high speed.

Chromatography is the process of separating a mixture by the distribution of its components between two phases, one phase stationary and the other phase moving. Some examples of chromatography are gas chromatography, paper chromatography, and thin-layer chromatography.

In this experiment you will separate a three-component mixture (containing sodium chloride, ammonium chloride and silicon dioxide) into the pure individual components. Knowing the mass of the original mixture and determining the mass of the pure components will allow you to calculate the percent by mass of each substance in the original mixture.

The separation scheme used to separate the mixture is based on differences in the physical properties (such as boiling point, melting point, solubility in a given solvent, etc.) of the three components.

Chemists frequently illustrate a separation procedure by means of a flow chart as depicted in the prestudy. By looking up the physical properties of each component in the mixture, they can decide what physical separation techniques will best allow them to separate the mixture. In the Prestudy for this experiment you will look up the pertinent physical properties of the three components and create a flow chart that outlines the separation procedure.

PROCEDURE

Obtain an unknown solid mixture from the instructor.

1. Separation of NH_4Cl. Weigh a clean, dry, evaporating dish and accurately weigh the ***total solid*** mixture into it. Place the evaporating dish on a hot plate ***in the hood.*** With the hot plate on high, heat the mixture until the white fumes cease. **Be patient. Depending on the amount of ammonium chloride in your sample, this could take quite a while. One hour would not be unheard of.** Stir the mixture frequently to facilitate the sublimation of the ammonium chloride. This is crucial in order to obtain good results. If all the ammonium chloride is not sublimed at this point, your other results will also be off. After the white fumes disappear, continue heating the mixture until ***no white solid (NH_4Cl) condenses on a stirring rod held above the evaporating dish.*** At this point all the NH_4Cl should be sublimated. Allow the evaporating dish to cool and record its mass to find the mass of NH_4Cl in the mixture.

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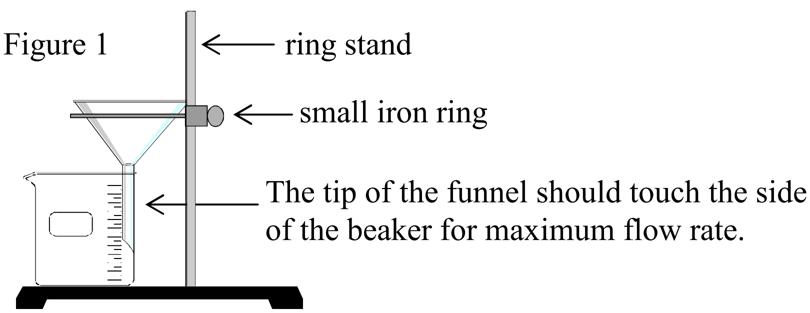
2. Extraction of NaCl. Weigh a 400 or 600 mL beaker. Weigh a piece of filter paper. Add between 5 to 7 mL of distilled water to the NaCl-SiO_2 mixture and stir gently for 3 minutes. Following the procedure outlined by the instructor (figure 1 below), filter the sand-salt solution through the weighed filter paper in the funnel into the weighed beaker. The solution that comes through the funnel should be completely clear. (Do not stir or poke the sand in the funnel; you may rip the fragile, wet filter paper.) Add 5 to 7 mL more distilled water to the evaporating dish to remove the rest of the sand and pour this through the funnel. Add another 5 to 7 mL distilled water to the evaporating dish and pour this through the funnel. If all the sand is now removed from the evaporating dish, pour the fourth 5 to 7 mL portion of water directly on the sand in the funnel. If the sand is not all out of the evaporating dish, pour the last portion of water into it to remove the rest of the sand, making sure that all the water and sand get into the funnel.

Figure 1



3. Drying Of SiO_2. Carefully place your wet filter paper cone in which the sand is contained onto a watch glass. There is no need to unwrap the cone. The instructor will then place the watch glass into a hot oven; do not go into the oven yourself. After approximately 30 minutes, the water should have all evaporated from the sand. The instructor will check and remove the dry sand and filter paper for you; do not go into the oven yourself Allow the paper and sand to cool and then weigh them to find the mass of SiO_2 in the sample.

4. Drying of NaCl. While the SiO_2 is in the oven, set the beaker containing the NaCl solution on a hot plate. Gently heat the solution until the salt is dry. (Start at a medium to high setting and then decrease the setting as the water evaporates.) Make sure that all the condensation is gone from the sides of the beaker. If the salt appears to be dry but there is still water on the beaker walls, see the instructor. If the salt pops out of the beaker, carefully remove the beaker from the hot plate and see the instructor. When the beaker is cool (check the bottom), weigh it to find the mass of NaCl in the original mixture.

5. Percent by Mass. Determine the percent by mass of ***each component*** in your mixture using the following formula:

$$\text{\% of Component A} = \frac{\text{grams of component A}}{\text{grams of original sample}} \times 100$$

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DATA AND CALCULATIONS

Unknown Number ______________________

1. Mass of evaporating dish ______________________
2. Mass of evaporating dish plus original sample ______________________
3. Mass of original sample ______________________
4. Mass of evaporating dish plus NaCl and SiO_2 ______________________
5. Mass of NH_4Cl in original sample ______________________
6. Mass of filter paper plus dry SiO_2 ______________________
7. Mass of dry filter paper ______________________
8. Mass Of SiO_2 in original sample ______________________
9. Mass of beaker plus dry NaCl ______________________
10. Mass of beaker ______________________
11. Mass of NaCl in original sample ______________________

Instructor's Initials ________

Calculations: Show all work!

1. Percent NH_4Cl in original sample

2. Percent SiO_2 in original sample

3. Percent NaCl in original sample

4. Percent lost

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PRESTUDY (page 1 of 2)

1. (5) Using the CRC Handbook of Chemistry and Physics or other suitable references, look up the following physical properties of ammonium chloride (NH_4Cl), silicon dioxide (SiO_2, sand, quartz), and sodium chloride (NaCl, table salt).

Substance	Formula	Melting Point (°C)*	Solubility†	Appearance
sodium chloride	NaCl			
ammonium chloride	NH_4Cl			
silicon dioxide (crystabolite)	SiO_2			

*Do not use the triple point (tp) for ammonium chloride; use the sublimation point (sp).
†Solubility: soluble (s) or insoluble (i) in water

2. (4) A student is given a 3.589 g mixture of iron filings, calcium chloride and sand. She separates the mixture and recovers 0.897 g of iron, 0.923 g of sand and 1.686 g of calcium chloride. Calculate the percentage of each of the three components she recovered from the original mixture and the percent of material she lost during the separation process.

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PRESTUDY (page 2 of 2)

3. (l) Using the above physical properties and the **procedure**, complete the following flow chart by giving the reagents and/or conditions necessary (in the ovals) to affect each indicated separation step and how the components (in the boxes) will be separated.