General Chemistry I
CHEM-1030
Laboratory Experiment No. 2
Physical Separation Techniques

Introduction

When two or more substances that do not react chemically are blended together, the components of the mixture retain their individual identity and properties. The separation of the components of a mixture is a problem frequently encountered in chemistry. The basis of separation theory is the fact that components of a mixture have different physical and chemical properties. The components are pure substances, 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 used for separation of mixtures include:

Distillation: The purification of a liquid by heating it to its boiling point, causing vaporization, and then condensing the vapor and collecting the liquid. Nonvolatile solids are easy to remove from a solution because they do not vaporize and are left behind during distillation. Separation of a mixture of two liquids requires that they have different boiling temperatures. Decreasing the pressure over the liquid will reduce all boiling temperatures.

Extraction: The removal of one substance from a mixture by use of a solvent that will dissolve one component but not the other.

Filtration: The process of removing or "straining" a solid (sometimes called a precipitate) from a liquid by the use of filter paper or other porous material.

Decanting: Pouring a liquid from a solid-liquid mixture, leaving the solid behind.

Sublimation: The physical process by which some substances can 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 sublimes, 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 substances that sublime easily.

Centrifuging: The process of separating a suspended solid from a liquid by spinning the mixture at high speed.

Chromatography: The separation of a mixture by the distribution of its components between a stationary phase and a moving phase. Some examples are gas chromatography, paper chromatography, and thin-layer chromatography.

In this experiment you will separate a three-component mixture of sodium chloride, iron filings, and silicon dioxide into its individual components. Measuring the mass of the original mixture and measuring the masses of the pure components will allow you to calculate the percent by mass of each substance in the original mixture.

The scheme used to separate the components of a mixture is based on differences in the physical properties of the three components. Chemists frequently illustrate a separation procedure with a flow chart like the one depicted in the Prestudy. By knowing the physical properties of each component, they decide what physical separation techniques will allow them to separate the mixture. In this experiment's Prestudy, you will look up the pertinent physical properties of some three components of a mixture and create a flow chart describing the separation procedure.

Safety

Chemical splash goggles must be worn at all times during this and all chemistry experiments, from the very beginning to the very end of the time you spend in the laboratory.

Disposal

Dispose of the iron in the heavy metal waste container. You may rinse sodium chloride residues down the drain with water. Dispose of the sand you isolate from the mixture in the regular trash can.

Cleanup

At the end of the lab period, wipe down all your work surfaces with a damp sponge.

Experimental Procedure

1. Obtain an unknown solid mixture and **record its number**.

2. Using a weigh-boat, determine the mass of your unknown solid mixture to the nearest 0.001g

A. Separation of Iron Filings. Place the bar magnet inside a sandwich bag. While holding one end of the magnet, move the other end through the mixture. The iron filings will stick to the magnet. Shake gently to remove any trapped sand. It is not necessary to remove all the iron filings at one time.

Gently lift the magnet with the iron filings and place the end with the filings inside a **preweighed 100-mL beaker**. Fold the plastic bag over the beaker. Carefully pull the magnet out of the bag, leaving the filings in the beaker.

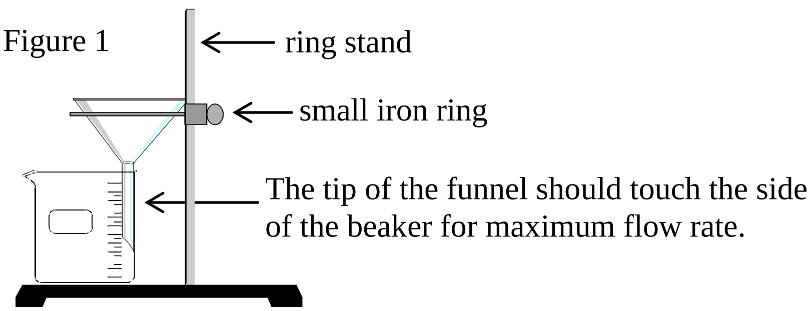
Repeat twice to remove the last traces of iron filings from the sand-salt mixture.

Determine the mass of the 100-mL beaker with the iron filings to the nearest 0.001 g.

Calculate the mass of NH_4Cl in the mixture.

B. Extraction of NaCl. Weigh a 400 or 600 mL beaker together with a watch glass to 0.001 g. Weigh a piece of filter paper to 0.001 g. Add about 5 mL of distilled water to the NaCl-SiO_2 mixture in an evaporating dish and stir gently for 1 minute. Assemble the setup shown in Figure 1 to filter the sand-salt solution through the weighed filter paper in a funnel into a weighed beaker. The solution that comes through the funnel should appear clear. (Do not stir or poke the sand in the funnel; you may rip the fragile, wet filter paper.) Add 5 mL more distilled water to the evaporating dish to dislodge the remaining sand and pour this through the funnel. Add another 5 mL of distilled water to the evaporating dish and pour this through the funnel. If all the sand is now gone from the evaporating dish, pour a fourth 5 mL portion of water directly onto the sand in the funnel. If any sand remains in the evaporating dish, pour the last portion of water into the dish to remove the rest of the sand, making sure all the water and sand go in the funnel.

Figure 1



C. Drying of SiO_2. Carefully place your wet filter paper cone with sand on a watch glass. There is no need to unwrap the cone. (Label or otherwise identify your filter paper or watch glass so they will not get mixed up with those of other students.) Place the watch glass in the laboratory drying oven. After about 30 minutes, the sand will be dry. Remove the watch glass, dried filter paper and sand from the oven. Allow them to cool to room temperature and weigh the filter paper and sand on the same balance you used earlier to weigh the filter paper. Calculate the mass of SiO_2 alone.

D. Drying of NaCl. While the SiO_2 is in the drying oven, place the beaker containing the NaCl solution on a hot plate. Place the watch glass on top of the beaker. Heat the solution to evaporate the water using a medium to high setting. At some point, the salt-water mixture will sizzle and pop, but the watch glass will keep all the sodium chloride inside the beaker. Make sure all condensation evaporates from the sides of the beaker. If the salt appears to be dry but there is still water on the beaker walls, continue heating until all the water is gone. Remove the beaker from the hotplate and let it cool to room temperature. Weigh the beaker, watch glass and salt together on the same balance you used to weigh the beaker and watch glass. Subtract to find the mass of NaCl in the original mixture. You may rinse the solid NaCl residue down the drain.

E. Percent by Mass. Determine the percent by mass of each component on your mixture using the following formula:

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

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Data page

Unknown Number ______________________

1. Mass of original sample ________________________
2. Mass of 100-mL beaker ________________________
3. Mass of 100-mL beaker + iron filings ________________________
4. Mass of iron filings in original sample ________________________
5. Mass of 400- or 600-mL beaker and watch glass ________________________
6. Mass of dry filter paper ________________________
7. Mass of filter paper plus dry SiO_2 ________________________
8. Mass Of SiO_2 in original sample ________________________
9. Mass of 400- or 600-mL beaker, watch glass, and dry NaCl ________________________
10. Mass of NaCl in original sample ________________________

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Report and Calculations

For full credit, show all your calculation setups clearly. All answers must contain the correct units and the correct number of significant figures.

1. Calculate the percentage of iron filings in the mixture.

2. Calculate the percentage of silicon dioxide in the mixture.

3. Calculate the percentage of sodium chloride in the mixture.

4. Calculate the percentage loss of the mixture in two ways:

a) Calculate the percentage lost by subtracting the three component loss percentages from 100%.

b) Calculate the percentage lost directly from the mass lost. The mass lost is the difference between the mass of the original mixture and the sum of the masses of the recovered components of the mixture.

5. Would incomplete drying increase or decrease the apparent percent value of the sand? Explain your answer.

6. Would incomplete extraction of iron filings increase or decrease the apparent percent value of iron filings? Explain your answer.

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Prestudy (first of two pages)

1. Use the CRC Handbook of Chemistry and Physics or other suitable reference on-line or print references to look up the physical properties of ammonium chloride (NH_4Cl), silicon dioxide (SiO_2, sand, quartz), and sodium chloride (NaCl, table salt) and enter them in the table. (4 points)

Substance	Formula	Melting Point (°C)*	Solubility[†]	Appearance
sodium chloride	NaCl			
ammonium chloride	NH_4Cl			white powder
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. 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 recovered from the original mixture and the percent of material *lost* during the separation process. (4 points)

PRESTUDY (second of two pages)

3. Using the physical properties you entered in the table and the outlined experimental procedure, complete the flow chart. In the oval spaces, name the reagents and/or conditions necessary to affect each indicated separation step. In the rectangular boxes, write the names of the separated components. (2 points)