Lab 1: Separation and Recovery of Components in a Ternary Mixture

Objectives:	To separate a mixture of silicon dioxide (sand), sodium chloride (table salt), and calcium carbonate; determine the mass percent of each component in the original mixture; and calculate the total recovery as a percentage of the original sample.
Materials:	A mixture of silicon dioxide (SiO_2), sodium chloride (NaCl), and calcium carbonate ($CaCO_3$); 3 M hydrochloric acid (HCl); 1 M potassium carbonate (K_2CO_3).
Equipment:	Two dry 150 mL beakers; 400 mL beaker; evaporating dish; stirring rod; filtering funnel and filter paper; iron ring, ring stand, and wire gauze pad; rubber policeman; hot plate; tongs; graduated cylinders; Büchner funnel and vacuum filtration apparatus.
Safety:	HCl is a corrosive solution and can cause burns. Handle hot glassware with tongs. Safety goggles should be worn at all times.
Waste disposal:	All recovered materials should be placed in the collection beaker; unused reagents and recovered solutions may be flushed down the drain with plenty of tap water.
Review:	You should be familiar with techniques for measuring masses and solution volumes and for filtering solutions. You should know that materials possess different characteristic properties, such as solubility and reactivity, and boiling points. You should know how to interpret chemical equations and use equations to represent chemical reactions.

Introduction

All matter exists as either pure substances or mixtures. A *pure substance* consists of only one kind of matter (element or compound), while a *mixture* consists of a combination of two or more substances. The components in a mixture retain their chemical identities, and the individual components of the mixture can be separated from one another and collected. In this exercise you will start with a *ternary* mixture (i.e., containing three components). After separating the mixture into its individual components, you will calculate the percent composition for each component as well as the total percent recovery of the original sample.

Separating Components of a Mixture

Mixtures may contain substances in various states: solid, liquid, or gas. To separate one component from the mixture, chemists take advantage of differences in the properties of the components. The two basic methods for separating mixtures are *physical* methods, which utilize differences in physical properties (solubility, boiling point), and *chemical* methods, which utilize differences in chemical reactivity. Suppose we wish to separate a solid from a liquid. One physical method useful in this case is to *decant* the liquid from the solid. The solid is allowed to settle to the bottom of the container. The liquid portion, called the *supernatant*, is then carefully poured off without disturbing the solid.

Another physical method is *filtration*. The mixture is poured through a porous material, such as filter paper. The liquid portion passes through the filter paper and is called the *filtrate*, while the solid portion is unable to pass through and is collected. The collected solid is called the *residue*. There are two common types of filtration. *Gravity filtration* is performed using a filtering funnel; gravity draws the liquid through filter paper, which is placed in the funnel. In *vacuum filtration*, an applied vacuum is used to draw the liquid through filter paper, which is held in a Büchner funnel.

In a *solution*, one component is dissolved in another and they cannot be separated by the physical methods discussed previously. Another physical method, such as heating, can be used. The liquid component is removed by *evaporation* and the solid component will remain in the container. If we have a mixture of two solids, they can be separated by *extraction* by adding a solvent in which only one of the solid components will dissolve. The insoluble component can then be separated by either decanting or filtration.

Mixtures can also be separated by chemical methods that involve selective reaction of one of the components to form a new substance. The physical properties of the new substance can then be used to separate it from the mixture. Once separated, a second reaction converts the new substance back to the original component. Consider the chemical and physical properties of the substances in the following ternary mixture (**Table 1.1**):

Table 1.1 Solubilities of Components in a Sample Ternary Mixture

Substance	Formula	Soluble in Water	Reacts with 3 M HCl
Silver chloride	AgCl	No	No
Potassium bromide	KBr	Yes	No
Nickel(II) carbonate	$NiCO_3$	No	Yes

Problem 1. How would you separate a mixture of AgCl and water?

Solution. Since the AgCl is insoluble in water we can isolate the solid AgCl by either decanting or filtration, as described previously.

Problem 2. How would you separate a mixture of KBr in water?

Solution. Since the KBr is soluble in water we cannot use the same approach. We can, however, heat the solution to evaporate the water and collect the solid KBr that remains.

Problem 3. How would you separate a mixture of solid KBr and solid AgCl?

Solution. The KBr is soluble in water while AgCl is not. We can extract the KBr by dissolving it in water and either decanting or filtering the solution to recover the solid AgCl. The solid KBr can then be recovered by evaporating the water from the filtrate.

Problem 4. How would you separate a mixture of solid AgCl and solid $NiCO_3$?

Solution. The $NiCO_3$ reacts with HCl but AgCl does not. By adding HCl to the mixture we can convert the $NiCO_3$ to $NiCl_2$ as shown in the following equation.

$$NiCO_3(s) + HCl(aq) \rightarrow NiCl_2(aq) + CO_2(g) + H_2O(l)$$

The insoluble AgCl can be recovered by filtration. The filtrate contains the dissolved $NiCl_2$, which can be converted back into the original $NiCO_3$ by adding K_2CO_3, and the solid $NiCO_3$ can be recovered by filtration.

$$NiCl_2(aq) + K_2CO_3(aq) \rightarrow NiCO_3(s) + 2KCl(aq)$$

Separation and Recovery of Components in our Ternary Mixture

The components of our ternary mixture are silicon dioxide (sand), sodium chloride (table salt), and calcium carbonate. The physical and chemical properties of these substances are summarized in **Table 1.2**.

Table 1.2. Selected Physical and Chemical Properties of Ternary Mixture Components

Substance	Soluble in Water	Reacts with 3 M HCl
SiO_2	No	No
NaCl	Yes	No
$CaCO_3$	No	Yes

The steps required to separate and recover the components of this mixture are outlined in **Figure 1.1**. First the NaCl can be extracted from the mixture by dissolving it in water; after filtering the insoluble SiO_2 and $CaCO_3$, the aqueous NaCl can be recovered from the filtrate by evaporation. The residue from the filtration can then be treated with HCl, which reacts with the $CaCO_3$ as shown

$$CaCO_3(s) + 2\ HCl(aq) \rightarrow CaCl_2(aq) + CO_2(g) + H_2O(l)$$

We can now decant the supernatant liquid to isolate the unreacted SiO_2. Boiling the supernatant and adding 1 M K_2CO_3 solution will precipitate the aqueous calcium ions as $CaCO_3(s)$, which can be recovered by filtration:

$$CaCl_2(aq) + K_2CO_3(aq) \rightarrow CaCO_3(s) + 2\ KCl(aq)$$

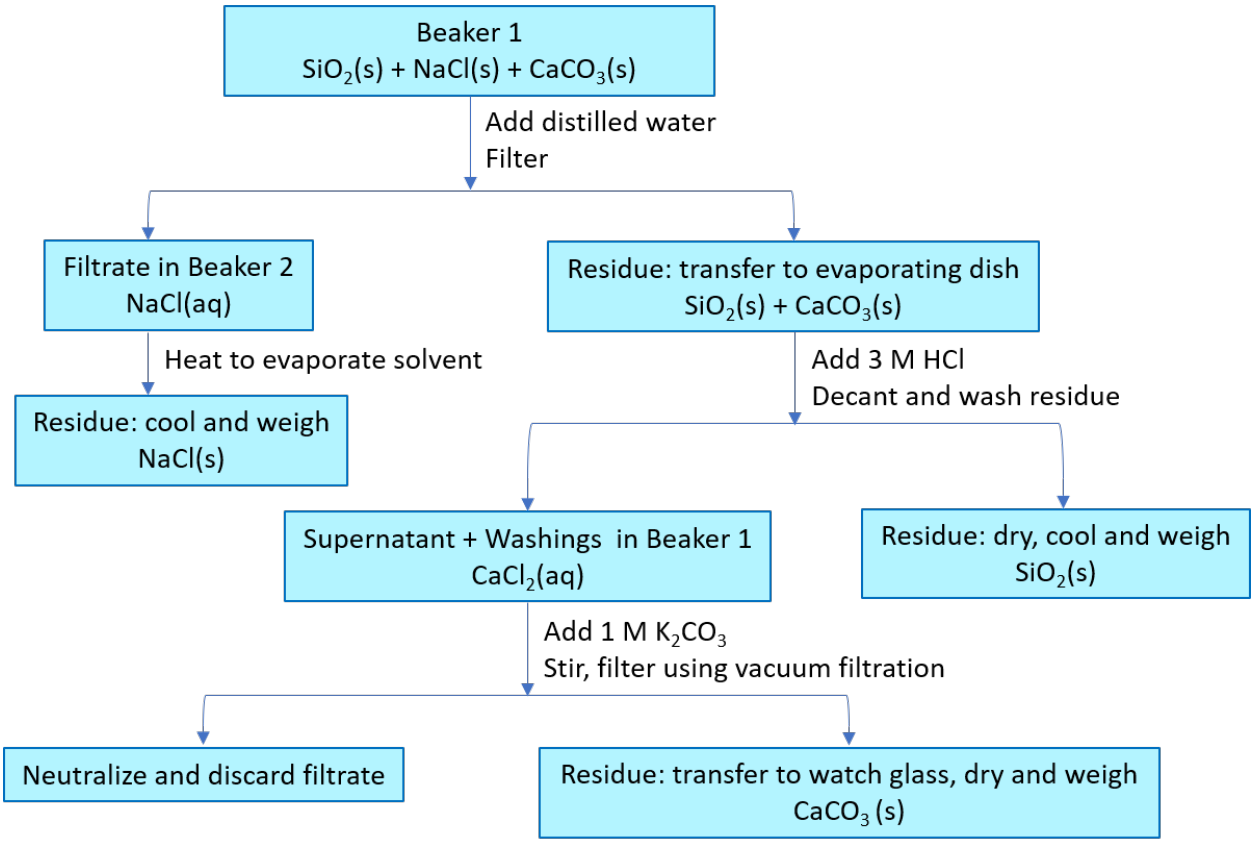

Figure 1.1. Flowchart for separation and recovery of components in a mixture of SiO_2, NaCl, and $CaCO_3$.

After drying and weighing each of the recovered components, we can calculate the mass percent of each component in the original mixture as shown in **Equation 1.1**:

Equation 1.1. $$\text{percent component in mixture (\%)} = \frac{\text{mass of recovered component (g)}}{\text{mass of original sample (g)}} \times 100\%$$

Finally, we use Equation 1.2 to calculate the total recovery of all components:

Equation 1.2. $$\text{total percent recovery} = \frac{\text{total mass of all recovered components (g)}}{\text{mass of original sample (g)}} \times 100\%$$

Ideally, the total percent recovery will be close to 100%. The efficiency of separation and recovery steps will vary, and losses of the individual components can occur and result in less than 100% recovery. Sample calculations are provided in the following example.

Problem 5. A KBr-AgCl-$NiCO_3$ mixture weighs 3.27 g. After separating the individual components, we recover 1.32 g KBr, 1.24 g AgCl, and 0.62 g $NiCO_3$.

Calculate the percent of AgCl in the original mixture:

$$\frac{1.24\text{ g AgCl}}{3.27\text{ g mixture}} \times 100\% = 37.9\%$$

Calculate the total percent recovery of the mixture:

$$\frac{1.32\text{ g}+1.24\text{ g}+0.62\text{ g}}{3.27\text{ g}} \times 100\% = 97.2\%$$

*Name:*_____ *Date:*_____

*Section:*_____

Pre-Lab Questions

1. List the hazards of dealing with 3 M HCl and describe the appropriate cautions to be taken when handling this material.

2. Define the following terms:
   a. decant

   b. filter (verb)

   c. extract

   d. filtrate

   e. supernatant

3. The following table lists properties for three solids.

Solid	**Soluble in Water**	**Reacts with 3 M HCl**
KBr	yes	no
$Mg(OH)_2$	no	yes
$BaSO_4$	no	no

   a. Describe the steps you would use to separate a mixture of solid KBr and $BaSO_4$ from each other.

   b. Describe the steps you would use to separate a mixture of solid $Mg(OH)_2$ and $BaSO_4$ from each other.

4. You are given a 4.32 g sample of a mixture of cobalt nitrate, magnesium sulfate, and naphthalene. After separation, you recover 2.34 g of cobalt nitrate, 0.25 g of magnesium sulfate, and 1.14 g of naphthalene.

   a. What was the mass percent of magnesium sulfate in the original mixture? Show your work!

   b. What was your total percent recovery?

5. Suppose you wish to separate a mixture of fine particles of gold and sand, neither of which is soluble in water. If you add liquid mercury to the mixture, the sand will float to the top of the mixture while the gold will dissolve. The sand can then be skimmed from the surface. You can recover the gold by heating the solution to evaporate the liquid mercury. Create a flow chart to illustrate this separation and recovery scheme.

Procedure

Observe all safety precautions when working with sample components and reagents.

Preparing Mixture for Separation

1. Label two dry 150 mL beakers 1 and 2. Measure and record the mass of each beaker on the **Data Sheet**.
2. Obtain a mixture of SiO_2, NaCl, and $CaCO_3$ from your lab TA. Record the ID code of your mixture (if any).
3. Transfer between 2.50 and 3.00 g of your mixture to beaker 1. Measure and record the mass of the beaker plus sample 1.

Separating and Recovering NaCl

4. Obtain 50 mL of distilled or DI water in a graduated cylinder. Add the water to the mixture in beaker 1 and stir for at least 2 min. to dissolve all the NaCl.
5. Have your TA demonstrate how to fold a piece of filter paper. Place a filtering funnel in a small iron ring in a ring stand as illustrated in **Figure 1.2**. Place a properly folded piece of filter paper in the funnel.

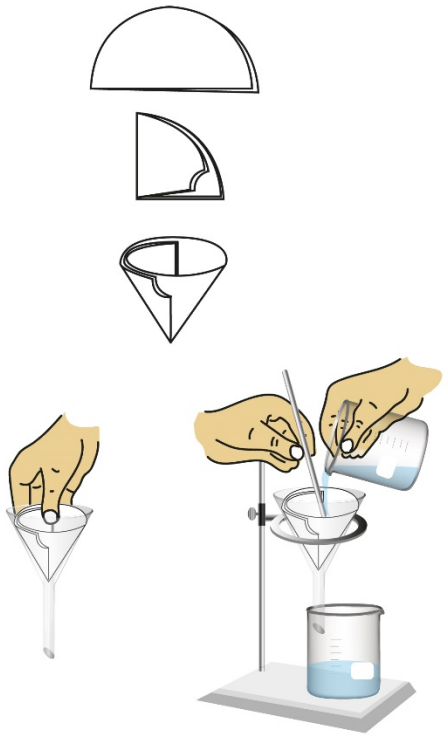

Figure 1.2. Gravity filtration technique.

6. Place beaker 2 under the funnel with the stem touching the inside wall of the beaker. Moisten the filter paper with distilled water from a wash bottle. Press the damp filter paper against the funnel so that it adheres tightly to the funnel wall.
7. Decant as much of the supernatant liquid as possible to the filter funnel, using a stirring rod to guide the solution as shown in **Figure 1.2**.
8. Once the supernatant has drained, transfer the solid residue to the filter paper using a rubber policeman.
9. Wash any remaining residue from beaker 1 onto the filter paper using a wash bottle. Save the filter paper and solid residue for use in **Part III**.
10. Place beaker 2 (with filtrate) on a hot plate and heat the solution to boiling. Once boiling begins, reduce the heat to maintain a gentle boil. Continue boiling until 3–5 mL of solution remains and adjust the heat to the lowest setting. Continue heating until all the liquid has evaporated.
11. Turn off the hot plate. Using tongs, carefully remove the beaker from the hot plate and place it on a ceramic-coated wire gauze. Allow the beaker to cool to room temperature. Measure the mass of beaker 2 plus residue and record this mass on the **Data Sheet**.
12. Wash, rinse, and dry both beakers for use in **Part III**.

Separating and Recovering SiO_2

13. Measure and record the mass of a dry evaporating dish.
14. Using forceps or tweezers, carefully remove the filter paper with residue recovered in step 9 from the filter funnel. Place the paper and residue in the evaporating dish. Carefully unfold the filter paper and wash the residue from the paper into the evaporating dish with about 5 mL of distilled water from the wash bottle. Discard the filter paper.
15. Obtain 8 mL of 3 M HCl in a clean graduated cylinder. Slowly add the acid to the residue in the evaporating dish. Adding the acid too quickly can result in the sudden release of gas and may cause losses of SiO_2 and $CaCO_3$. Stir the solution until all the gas has been released and bubbles stop forming in the reaction solution.
16. Decant as much of the supernatant liquid as possible from the evaporating dish into beaker 1. Wash the solid residue in the dish with 5 mL of distilled water from your wash bottle. Allow the solid to settle and decant the wash water into beaker 1. Repeat the washing and decanting steps twice using 5 mL of distilled water each time and collect all washings in beaker 1. Save the supernatant plus washings for use in step 19.
17. Place the evaporating dish and residue on the ceramic-coated wire gauze and carefully transfer it to the oven to dry. Continue heating the evaporating dish until the solid residue is completely dry. Remove from oven using tongs and place it on a ceramic-coated wire gauze.

18. Allow the evaporating dish and contents to cool to room temperature. Measure and record the mass of the dish plus residue. Discard the SiO_2 as instructed by your TA.

Recovering $CaCO_3$

19. Place beaker 1 with supernatant and wash solutions (step 16) on a hot plate. Heat the solution to boiling and allow it to boil for 5 min. While the solution is heating, obtain 15 mL of 1 M K_2CO_3 in a clean graduated cylinder. After the supernatant has boiled for 5 min., use tongs or paper towels to transfer beaker 1 from the hot plate to a ceramic-coated wire gauze pad. Immediately add the 1 M K_2CO_3, and stir the reaction mixture for 5 min. Allow the mixture to cool to room temperature.
20. Label a dry watch glass with an identifying mark. Place a piece of filter paper for a Büchner funnel on the watch glass. Measure and record the mass of the watch glass plus filter paper.
21. Assemble a vacuum filtration apparatus as indicated in **Figure 1.3**. Place the pre-weighed piece of filter paper in the Büchner funnel and moisten with a few drops of distilled water.

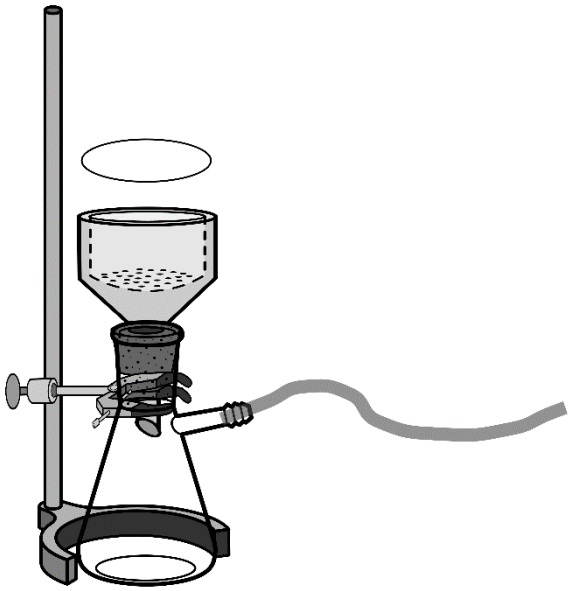

Figure 1.3. Büchner funnel vacuum filtration apparatus.

22. Turn on the water aspirator.
23. Decant the supernatant liquid in beaker 1 to the Büchner funnel, using your stirring rod to guide the liquid from the beaker onto the filter paper. Use a rubber policeman to transfer the solid residue from the beaker to the filter paper in the Büchner funnel.
24. Rinse any remaining solid from beaker 1 to the filter paper using water from your wash bottle. Continue to draw air through the filter paper for at least 5 min. Turn off the water aspirator, remove the Büchner funnel, and discard the filtrate.

25. Use forceps or tweezers to carefully remove the filter paper with the residue from the Büchner funnel. Place the filter paper and residue on the pre-weighed watch glass. Place the watch glass and filter paper/residue in the oven (refer to step 17). Continue heating until the filter paper and residue are dry. Use tongs or paper towels to transfer the watch glass and contents from the oven to a ceramic-coated wire gauze.
26. Allow the watch glass and contents to cool to room temperature. Measure and record the mass of the watch glass plus contents on the data sheet. Discard the $CaCO_3$ as instructed by your TA.

Repeat the Determination

27. Perform a second separation by repeating steps 1 and 3–26 (if time permits).

*Name:*_____ *Date:*_____

*Section:*_____

Data Sheet

ID code of mixture: _____

Sample	Determination 1	Determination 2
Mass of beaker 1 + sample (g)		
Mass of beaker 1 (g)		
Mass of sample (g)*		
Mass of beaker 2 + NaCl (g)		
Mass of beaker 2 (g)		
Mass of NaCl (g)*		
Mass of evaporating dish + SiO_2 (g)		
Mass of evaporating dish (g)		
Mass of SiO_2 (g)*		
Mass of watch glass + filter paper + $CaCO_3$ (g)		
Mass of watch glass + filter paper		
Mass of $CaCO_3$ (g)*		

*determined as the difference of prior mass measurements

Data and Calculations

ID code of mixture: _____

Copy your mass calculations from the data sheet to the appropriate location below.

Sample	Determination 1	Determination 2
Mass of sample (g)*		
Mass of NaCl (g)*		
% NaCl in mixture (%)		
Mass of SiO_2 (g)*		
% SiO_2 in mixture (%)		
Mass of $CaCO_3$ (g)*		
% in $CaCO_3$ mixture (%)		
Total mass of recovered components (g)		
Total percent recovery (%)		

Show sample calculations below:

*Name:*_____ *Date:*_____

*Section:*_____

Post-Lab Questions

1. Answer the following questions related to the laboratory procedure:

   a. In step 16 of the procedure you separated the solid SiO_2 by decanting the supernatant liquid. Provide an alternative method for this separation.

   b. After decanting the supernatant, you washed the solid three times. Explain why these washings were necessary.

2. Your total percent recovery may not be 100%.
   a. Describe a source of error that could result in a percent recovery of less than 100%.

   b. Describe a source of error that could result in a percent recovery of more than 100%.

3. Based on your observations, speculate on which of the three components was the most difficult to separate and recover. Briefly explain your selection.

4. Based on the mass percentages for your sample, describe how much of each component you would use to prepare a 5.00 g sample of your mixture.