# EXPERIMENT 2
## SEPARATING A MIXTURE OF CALCIUM CARBONATE, SALT, AND SAND

<u>Materials Needed</u>

Sample unknown
150 mL beakers (**2**)
400 mL beaker
10 mL graduated cylinder
50 mL or 100 mL graduated cylinder
Hot plate
Wash bottle
Labels
Large glass funnel
Coors evaporating dish
Rubber Policeman
Ring stand and ring
Stirring glass rod
Filter paper
Watch glass

<u>Background</u>

Purifying a substance from a mixture is central to human progress. You have no doubt heard of the ancient Bronze Age and later Iron Age, corresponding to times when metals could be isolated from ore to make better tools and weapons. Progress continues today for example, in manufacturing highly pure silicon for microchips and isolating medically active compounds from rainforest plants. All purification methods use the chemical or physical properties of the substance in order to isolate it from others in the mixture.

In this experiment you will separate a mixture of three common substances: calcium carbonate ($CaCO_3$), sodium chloride (NaCl), and sand. $CaCO_3$ is the main component of limestone, a common sedimentary rock. Sand is silicon dioxide ($SiO_2$) otherwise known as 'silica' or 'quartz sand'. NaCl is the most common ionic compound.

Since NaCl is soluble in water and the other two components in the mixture are not, NaCl is removed first by stirring the unknown mixture in water and pouring off, or **decanting**, the water solution on top after the solids of $CaCO_3$ and $SiO_2$ have settled. A solution above a settled solid is called a **supernatant**. The NaCl in the supernatant is isolated by boiling off the water. NaCl is soluble in water because the positive and negative ions of NaCl crystals dissociate and bond to water molecules according to Equation 1, where (s) represents solid, and (aq) represents **aqueous** (meaning 'dissolved in water').

$$NaCl_{(s)} \rightarrow Na^{+}_{(aq)} + Cl^{-}_{(aq)}$$

**Equation 1.** Representation of salt dissolving.

Figure 1 shows how water molecules interact with the ions of salt in solution.

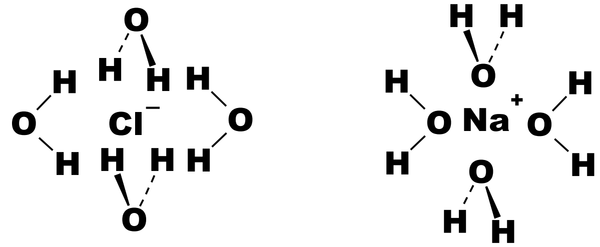


**Figure 1.** Water molecules can bond both to **cations**(positive ions) and **anions** (negative ions) so water dissolves many salts.

Notice in Figure 1 that the water molecules bond differently with the two ions. The oxygen atoms of water are attracted to the sodium cation ($Na^+$), whereas the hydrogen atoms of water are attracted to the chloride anion ($Cl^-$). The reason for this difference is because a water molecule is **polar**; the oxygen side of the water molecule is slightly negative whereas the hydrogen side is slightly positive.

When the water is boiled away Equation 1 is reversed and solid NaCl is obtained. Note this is a physical process, not a chemical process, since the salt merely dissolves and is then recrystallized.

After the salt has been removed from the mixture, the $CaCO_3$ and $SiO_2$ are separated by reacting the $CaCO_3$ with hydrochloric acid ($HCl_{(aq)}$) to dissolve the calcium ion as calcium chloride according to Equation 2.

$$CaCO_{3(s)} \;+\; 2\,HCl_{(aq)} \;\rightarrow\; CaCl_{2(aq)} \;+\; H_2O_{(\ell)} \;+\; CO_{2(g)}$$

**Equation 2.** Insoluble calcium carbonate reacts with hydrochloric acid to form soluble calcium chloride, water, and carbon dioxide gas.

where ($\ell$) represents liquid and (g) represents gas. The 'fizzing' you will observe is the carbon dioxide gas being formed. The $CaCl_2$ product of Equation 2 is dissolved in the form of separated $Ca^{2+}$ and $Cl^-$ ions bound to water molecules, in a similar fashion as the ions of salt illustrated in Figure 1.

The aqueous solution of calcium chloride ($CaCl_2$) then is decanted from the silica which settles to the bottom. In this case, you are exploiting a chemical property of $CaCO_3$ since it reacts with HCl to form a new compound, $CaCl_2$. Considerable carbon dioxide ($CO_2$) with fizzing is produced when reacted with $HCl_{(aq)}$. This method is used as a geologic field test for limestone. Sand is not affected by the acid since $SiO_2$ is almost completely **insoluble** (i.e. does not dissolve) in hydrochloric acid.

In order to retrieve the $Ca^{2+}$ ions from water solution and reform $CaCO_3$, the $CaCl_2$ solution is reacted with potassium carbonate ($K_2CO_3$) according to Equation 3.

$$CaCl_{2(aq)} \;+\; K_2CO_{3(aq)} \;\rightarrow\; CaCO_{3(s)} \;+\; 2\,KCl_{(aq)}$$

**Equation 3.** Calcium chloride is reacted with potassium carbonate to reform insoluble calcium carbonate.

The solid $CaCO_3$ obtained is filtered, washed, and dried. Washing and drying of the remaining sand completes the separation of the three component mixture.

In theory, you should get back all three components of the mixture so that their combined masses are the same as the starting mixture. However, when a difference occurs between actual results and what should be obtained in theory, the value of the **percentage error** for the experiment may be given according to the following equation.

$$\%\text{error} = \left|\frac{\text{actual} - \text{theoretical}}{\text{theoretical}}\right| \times 100\%$$

**Procedure**

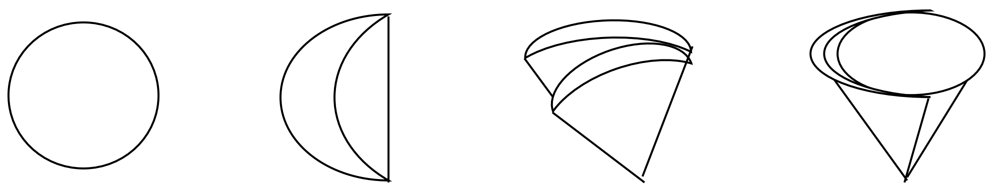
Purifying the NaCl

1. Obtain two clean 100 mL or 150 mL beakers and label them Beaker 1 and Beaker 2. Weigh each beaker to the nearest 0.0001 g and record their masses.

2. Obtain an unknown mixture and record the unknown number on your data sheet. Precisely weigh out between 2.5000 g and 3.0000 g of your unknown into Beaker 1 and record the mass.

3. Fill the wash bottle with deionized water. Use a graduated cylinder to add 50 mL of deionized water to Beaker 1 and swirl for two minutes or so to dissolve the NaCl.

4. Obtain a round filter paper and fold it in half; fold it in half again. Form a cone with the paper and place it in a glass funnel. The cone should be three thicknesses of paper on one half and one thickness on the other half.



**Making a cone of filter paper**

5. Use a support ring to hold the funnel over Beaker 2 and wet the filter paper with deionized water from a wash bottle. Press the filter paper against the wall of the funnel to form a tight seal. Make sure the tip of the funnel is near the bottom of Beaker 2.

6. Carefully pour the supernatant from Beaker 1 into the funnel using a stirring rod to guide the liquid from the beaker to the funnel. After the supernatant is poured off, carefully wash all of the solids of Beaker 1 into the funnel using a wash bottle and stirring rod with rubber policeman. Be careful not to overfill the funnel with water.

7. After almost all of the supernatant has passed through the funnel, rinse around the top of the filter paper using no more than 50 mL of deionized water from the wash bottle and let the water drain through the funnel. Repeat the rinse and let the water drain through again. At this point, all of the NaCl solution has been transferred to Beaker 2.

8. Place the NaCl solution of Beaker 2 on a hot plate at a medium temperature setting. Keep an eye on the NaCl solution as it evaporates. When almost all of the liquid water is gone but the salt crystals are still damp, remove the beaker from the hot plate. The final drying of the NaCl crystals must be done slowly or the crystals will spatter. The beaker may be returned to the hot plate intermittently with care to complete the final drying. (Note: While you are waiting for the NaCl to dry, you should begin separating $SiO_2$ and $CaCO_3$ as described in the following section.)

9. When the NaCl is dry and Beaker 2 has cooled to room temperature, record the mass. (**Caution: Never weigh hot objects.**) Rinse out Beaker 2 in the sink.

Separating $SiO_2$ and $CaCO_3$

1. Record the mass of a clean, dry evaporating dish to the nearest 0.0001 g. Carefully remove the filter paper from the glass funnel, and scrape the solids of the filter paper into the evaporating dish with the rubber policeman. Use the wash bottle to wash the remaining solids on the filter paper into the evaporating dish. Discard the cleaned filter paper.

2. Measure 8 mL of 3 M HCl in a clean, dry small graduated cylinder. Slowly add the acid to the evaporating dish and stir with a clean stirring rod until gas fizzing stops.

3. When the sand has settled in the evaporating dish, decant the supernatant $CaCl_2$ solution into clean Beaker 1. Be sure that no sand is transferred from the evaporating dish to Beaker 1. Add 5 mL of deionized water to the sand in the evaporating dish and stir. After the sand has settled, pour the supernatant into Beaker 1. Repeat this washing of the sand with another 5 mL portion of deionized water and decant again.

4. Dry the $SiO_2$ by placing the evaporating dish on top of a 400 mL beaker of boiling water on a hot plate. After the $SiO_2$ is completely dry, remove the evaporating dish and let cool. Dry the bottom of the evaporating dish containing sand and record the mass on the data sheet. Discard the $SiO_2$ in the trash at the end of the experiment.

Purifying the $CaCO_3$

1. Place Beaker 1 on a hot plate and heat to boiling. Carefully remove Beaker 1 from the hot plate and immediately add 15 mL of 1 M $K_2CO_3$ solution. Stir the mixture for 2 minutes and set aside to allow the mixture to cool to room temperature.

2. Using a pencil, write your initials on a sheet of filter paper. Weigh the filter paper and the watch glass to the nearest 0.0001 g, fold it to make a cone and place it in a glass funnel. Wet the paper with a wash bottle.

3. Place an empty beaker under the glass funnel to catch the filtrate waste and carefully pour the supernatant of Beaker 1 into the funnel using a glass stirring rod to direct the liquid. Carefully wash all of the solids of Beaker 1 into the funnel using a squirt bottle and stirring rod with rubber policeman. After most of the supernatant has passed through the funnel, rinse the top of the filter paper with the squirt bottle and let it drain through the funnel. Repeat the rinse and let the water drain through again. Discard the filtrate liquid in the sink.

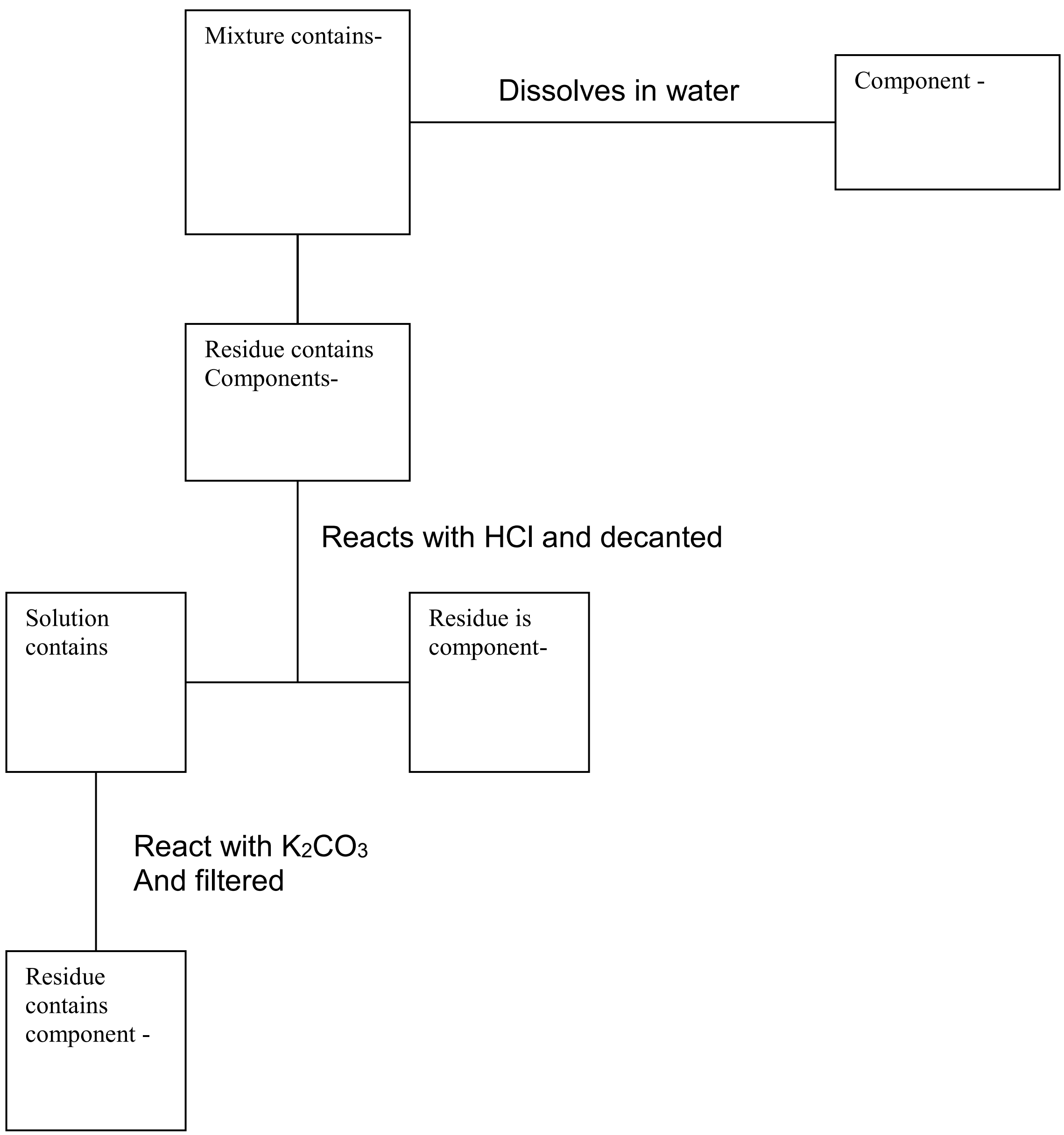
4. Remove the filter paper cone containing the $CaCO_3$ and place it on a watch glass. Dry the watch glass and filter paper containing $CaCO_3$ on top of a beaker of boiling water or in the oven in the back room of the laboratory. After the sample is completely dry, cool to room temperature and record the mass on the data sheet. **If the $CaCO_3$ is not dry at the end of the first period, leave it on the watch glass in your drawer to**

**dry until the second week of the experiment before weighing.** Discard the filter paper and $CaCO_3$ in the trash.

Mass Percent and Percent Recovery Calculations

1. Determine the mass percent of each component in the mixture by dividing the mass of each component by the mass of the whole sample used at the beginning of the experiment (and of course, also multiplying by 100). The percent recovery is calculated by dividing the total mass recovered by the mass of whole sample used at the beginning of the experiment.

Complete the following flow chart that describes the separation of this mixture.



**EXPERIMENT 2**

SEPARATING A MIXTURE OF CALCIUM CARBONATE, SALT, AND SAND

**Data Sheet** 40 Points

**Unknown Number**:____________ Name:_______________________

| | Determination First | Pt value |
|---|---|---|
| Mass of beaker **1** + sample, (g) | | 1 |
| Mass of beaker **1**, (g) | | 1 |
| Calculated mass of whole sample, (g) | | 2 |
| | | |
| Mass of beaker **2** + NaCl, (g) | | 1 |
| Mass of beaker **2**, (g) | | 1 |
| Calculated mass of NaCl, (g) | | 2 |
| | | |
| Mass of evaporating dish + $SiO_2$, (g) | | 1 |
| Mass of evaporating dish, (g) | | 1 |
| Calculated mass of $SiO_2$, (g) | | 2 |
| | | |
| Mass of filter paper + watch glass + dry $CaCO_3$, (g) | | 1 |
| Mass of filter paper + watch glass (g) | | 1 |
| Calculated mass of $CaCO_3$, (g) | | 3 |
| Total mass of recovered NaCl, $SiO_2$ and $CaCO_3$, (g) | | 3 |
| | | |
| Mass percent NaCl in the mixture | | 4 |
| Mass percent $SiO_2$ in the mixture | | 4 |
| Mass percent $CaCO_3$ in the mixture | | 4 |
| Percent Recovery | | 4 |
| Percent Error | | 4 |

**Show percentage calculations:**