Separation of the Components of a Mixture

Prepared by Edward L. Brown, Lee University

To become familiar with the laboratory techniques used to separate different substances from one another. Students will gain skill in the techniques of decantation and extraction.

**APPARATUS**

<table>
<thead>
<tr>
<th>Item</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>200 mL evaporating dish</td>
<td>(2)</td>
</tr>
<tr>
<td>12.5 cm watch glass</td>
<td>(2)</td>
</tr>
<tr>
<td>Bunsen burner and hose</td>
<td></td>
</tr>
<tr>
<td>wire gauze</td>
<td></td>
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<tr>
<td>beaker tongs</td>
<td></td>
</tr>
<tr>
<td>ring clamp</td>
<td></td>
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<tr>
<td>stirring rod</td>
<td></td>
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</tbody>
</table>

**CHEMICALS**

Unknown | (2.5 - 3 g)

Mixtures can be divided into two major classes – **homogeneous** and **heterogeneous**. A **homogeneous mixture** is composed of two or more pure substances that when mixed together have the physical appearance of uniformity. Salt water is an example of a homogeneous mixture of pure salt and pure water. Both salt water and pure water can appear identical – their difference is revealed upon tasting (or evaporating, etc.) the two solutions. A **heterogeneous mixture** contains two or more pure substances, but lacks the uniformity described above. Wet sand is an example of this type of mixture. Close inspection reveals a mixture of a liquid material with a solid material – uniformity, if it exists at all, is confined to small areas or pockets. In addition to these two broad categories, chemists also use the term “**impure substance**” when speaking of a mixture that overwhelmingly contains a single pure substance and small amounts of impurity.

The pure substances that form mixtures can be separated from one another through techniques that exploit both their physical and chemical properties. Physical properties unique to a pure substance are melting point, boiling point, refractive index, and density. In addition to these physical properties, a substance’s solubility in a particular solvent or the decomposition of a substance are physical and chemical properties that can be used to separate components of a mixture. The separation of the components in many mixtures routinely encountered by chemists is based on the use of one or more of the following techniques:

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Separation of the Components of a Mixture

1. **Decantation.** The process of separating a liquid from a denser solid. The solid component settles to the bottom of the container leaving the less dense liquid at the top where it is removed by pouring.

2. **Extraction.** The process of separating substances by exploiting their differing solubilities in a particular solvent. Ideally, only one component of a mixture will dissolve in the solvent of choice leaving the other component as a solid or an immiscible liquid.

3. **Filtration.** The process of separating a liquid from a solid by moving the solution through a material that is porous to the liquid but not the solid. Typically, filter paper and gravity are used to accomplish this separation.

4. **Crystallization.** The process of providing conditions in which only one substance in a solution will form crystals that can be collected by filtration.

5. **Distillation.** A process in which components of mixtures are separated based on differing boiling points – the lowest boiling component is collected first followed by the less volatile components of the mixture. The mixture is heated to its boiling point using a heating mantle. The thermometer records the temperature of the vapor and the condenser cools the vapor down to its liquid state where the angle of the condenser and gravity permits the liquid to drip into the receiving flask.

6. **Sublimation.** A process in which a solid passes directly into the gas state without becoming a liquid. All substances can accomplish this feat at some particular temperature and pressure, however, at atmospheric pressure relatively few substances sublime.

7. **Chromatography.** A process in which components of a mixture are exposed to two very different physical environments – a stationary phase and a mobile phase. Usually, two components in a mixture will not have the same affinity for both phases. The extent of separation depends on each component's time in the mobile phase – the longer a component is in this phase, the farther it will travel along the stationary phase. In Step 1, pure A,
Separation of the Components of a Mixture

pure B and a mixture of A & B are spotted at the bottom of the stationary phase. The mobile phase is allowed to pass through the components in Step 2 and carry them up the stationary phase. The stationary phase is withdrawn in Step 3 and the positions of A and B are located – note the separation between A and B that occurred in the third lane.

The mixture that you will separate contains three components: NH₄HCO₃ (ammonium bicarbonate), NaCl (salt), and SiO₂ (sand). The first step in the separation involves the decomposition of NH₄HCO₃ into ammonia, carbon dioxide and water. This will leave behind the NaCl and SiO₂. The NaCl is isolated by dissolving it in water and decanting the salt water away from the insoluble SiO₂ (sand). After evaporating the salt water to dryness and drying the sand, the masses of each component in the mixture will be known and their individual percentages can be determined [Equation 3.1].

Part of your grade is based upon how accurately you performed the experiment: are your percentages precise AND do the three percentages calculated in Step 24 add up to be 100%. More than 100% usually represents wet salt or sand at the end of the separation scheme (or student error). Less than 100% represents loss of material through spillage or spattering. The first problem can be remedied by reheating the evaporating dishes and reweighing. Therefore, it is advisable to keep the evaporating dishes, watch glasses, and their contents until you have added the three masses and made sure they are less than (or equal to) the mass of your original mixture.

A. Separation of Ammonium Bicarbonate from Salt and Sand

1. Record the Unknown Number [Data Sheet Q1].
2. Place a small beaker on the balance. Lower the draft controller and press the TARE button. Inside the beaker, place a clean, dry Pyrex test tube. Lower the draft controller and record its mass [Data Sheet Q2].
   • Record all the masses in today’s lab to the number of significant figures shown on the balance.
   • Also, if the reading on the balance is not stable, make sure you minimize drafts – otherwise, find another balance.
   • Finally, use the same balance throughout this lab.
3. Remove the test tube and add between 2.5 and 3.0 g of your Unknown Heterogeneous Mixture (HM).
   • First, shake the unknown in its container thoroughly and hold the test tube over the Unknown Container while adding the Unknown to the test tube.
   • Check the mass of the unknown regularly by gently placing the test tube / unknown into the beaker that is still sitting on the balance.
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- When the mass on the balance reads between 2.5 and 3.0 g higher than the mass of the empty test tube, wipe the outside of your test tube with a paper towel, place it in the beaker, lower the draft controller and accurately record the mass of the test tube and unknown [Data Sheet Q3].

4. Determine the mass of the original mixture [On-line Report Sheet Q4].
5. IN THE HOOD!! Hold your test tube with a clamp and heat it with a Bunsen burner until the solid has decomposed into its gaseous products: CO₂, NH₃ and H₂O.
   - This will take 5 – 10 minutes depending on the amount of NH₄HCO₃ present in your unknown and the temperature of the flame.
   - You will need to heat the entire length of the tube to completely remove the water that collects in the mouth of the test tube as steam.

6. Allow the test tube to cool and obtain its mass [Data Sheet Q5].
7. Determine the mass of the ammonium bicarbonate in the mixture [On-line Report Sheet Q6].
8. Obtain a large evaporating dish (100 – 200 mL capacity).
   - It should be free of dirt, water, labels, etc.
   - A clean dish is essential if you are to obtain accurate measurements since dirt and water and labels can come off during the experiment giving inaccurate mass differences.
   - If you need to clean the evaporating dish, use an abrasive cleanser, dry the dish with a paper towel and complete the drying process in a flame for ~30 seconds.
   - Also, obtain a watch glass that will fit on top of the evaporating dish – make sure it is clean and dry.

9. Record the mass of the Evaporating Dish and Watch Glass [Data Sheet Q7].
10. Add ~ 5 mL water to the test tube from Step 6.
    - Use a stirring rod to gently break up any solid clumps (don’t poke a hole in the bottom of the test tube).
    - Warm the solution by moving it back and forth in the flame of a Bunsen Burner for ~ 60 seconds – Do Not Heat To Boiling!!
    - Decant this solution to the Evaporating Dish – Do Not Transfer Any Sand During The Decantation!!

11. Repeat Step 10 with an additional 5 mL of water.
12. Repeat Step 10 with an additional 5 mL of water (15 mL water total).

B. Separation of Salt from Sand

13. Hold the test tube containing the wet sand with a test tube clamp and heat it gently with a Bunsen burner to remove the water.
14. Place the test tube in a beaker to cool for ~ 5 minutes.
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15. Weigh the test tube and its contents [Data Sheet Q8].
16. Record the mass of the sand in your unknown mixture [On-line Report Sheet Q9]
17. To remove the water from the aqueous salt solution, assemble the apparatus shown in Figure 3.1. Heat the evaporating dish with a Bunsen burner. **Take care to avoid loss of material – heating too strongly can cause the liquid to boil out of the dish.**
18. Once the water has completely evaporated (no water is present on the underside of the watch glass), remove the Bunsen burner and allow the evaporating dish / watch glass to cool.
19. When you can comfortably hold the dish in a paper towel, weigh the dish, watch glass, and its contents [Data Sheet Q10].
20. Record the mass of the salt in your unknown mixture [On-line Report Sheet Q11]
21. Discard the salt and sand in the regular trash receptacle.
22. Clean the test tube, stirring rod, evaporating dish and watch glass with soap and water, dry them and return them to the appropriate location.
23. Return the Bunsen burner and ironware to the appropriate location.

C. % Composition of Ammonium Bicarbonate, Salt and Sand


**Lab Report:** Once you have turned in your Instructor Data Sheet, lab attendance will be entered and lab attendees will be permitted to access the online data / calculation submission part of the lab report (click on Lab 3 – Separation of the Components of a Mixture). Enter your data accurately to avoid penalty. The lab program will take you in order to each calculation. If there is an error, you will be given additional submissions (the number and penalty to be determined by your instructor) to correct your calculation.

**Post-Lab Questions:** The questions for this lab can be found at [http://www.Chem21Labs.com](http://www.Chem21Labs.com). **Do Not Wait Until The Last Minute!!!! Computer Problems and Internet Unavailability Happen, But Deadlines Will Not Be Extended!!** On the Internet, complete the Post Lab Questions at the end of Lab 3 – Separation of the Components of a Mixture. The computer program will check your answers (with the correct number of significant figures) as they are entered. If there is an error, you will be given additional submissions (the number and penalty to be determined by your instructor) to correct your answer.

**Late Submission:** Late submission of the lab data / calculations is permitted with the following penalties: **- 10 points** for submissions up to 1 day late, **- 20 points** for submissions up to 2 days late.
Laboratory 3
Student Data Sheet

A. Separation of Ammonium Bicarbonate from Salt and Sand

1. Unknown Number

2. Mass of empty test tube

3. Mass of test tube, NH₄HCO₃, NaCl and SiO₂

5. Mass of test tube, NaCl and SiO₂

7. Mass of Evaporating Dish and Watch Glass

B. Separation of Salt from Sand

8. Mass of test tube and sand

10. Mass of Evaporating Dish, Watch Glass and salt

Laboratory 3
Instructor Data Sheet

A. Separation of Ammonium Bicarbonate from Salt and Sand

1. Unknown Number

2. Mass of empty test tube

3. Mass of test tube, NH₄HCO₃, NaCl and SiO₂

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7. Mass of Evaporating Dish and Watch Glass

B. Separation of Salt from Sand

8. Mass of test tube and sand

10. Mass of Evaporating Dish, Watch Glass and salt

Name: ____________________

Experiment 3 3-6