EXPERIMENT 4

Separation of a Mixture

INTRODUCTION

In this experiment you will be separating two components in a mixture by filtration, and recovering a dissolved component by evaporation.

Filtration is a technique for separating two components in a mixture if one of the components is a solid and the other component is a liquid. In this experiment, sodium chloride and sand will be separated using filtration because sodium chloride will dissolve in water (so it will now be in the liquid state) and sand remains in the solid state. The liquid, which is in contact with the solid, is called the supernatant. To separate the sand from the supernatant liquid, the mixture will be passed through filter paper in a funnel. The sand will be trapped by the filter paper, and the liquid will pass through. The liquid that will pass through the filter paper is called the filtrate. The filtrate will contain the dissolved sodium chloride, and the sodium chloride will be recovered by evaporating away the water. This will be done in a porcelain evaporating dish with a watch glass cover that prevents spattering.

Heated glassware, porcelain, and iron rings look cool several seconds after heating, but can still burn for several minutes. Use heat-safety items such as crucible tongs or beaker tongs whenever you handle this apparatus. You can test the temperature of questionable beakers, ring stands, wire gauze, or other pieces of apparatus that have been heated by holding the back of your hand close to their surfaces before grasping them. Any heat generated from the hot surfaces will be felt - do not touch. Allow plenty of time for the apparatus to cool before handling.

PROCEDURE

1. Students will work individually for this experiment. Except for the laboratory handout, remove all books, purses, and such items from the laboratory bench top, and placed them in the storage area by the front door. For laboratory experiments you should be wearing closed-toe shoes. Tie back long hair, and do not wear long, dangling jewelry or clothes with loose and baggy sleeves. Open you lab locker. Put on your safety goggles, your lab coat, and gloves.

2. Find your metal scoupla in your drawer along with a your evaporating dish and 100 mL beaker to the milligram balance.

3. Carefully open the side sliding door of the milligram balance. Watch for debris in the track of the sliding door and clean it out if it is present because the sliding door is one of the most fragile parts of the balance. Place a weighing cup on the balance pan and close the door. Press one of the keys with the symbol “$\rightarrow T/0 \leftarrow$” and make sure the display reads "0.000 g". When the display shows a mass with three places past the decimal point followed by a "g" for grams, carefully add between 0.300 and 0.400 g of sodium chloride to the weighing cup. Record the measurement in your Data Table. Record all digits shown.
**CAUTION:** Never place your microspatula or scoopula into a reagent bottle. You should pour a small amount of the reagent into a separate weighing cup by pouring and use your scoopula or microspatula to add to your weighing cup.

**CAUTION:** Never pour solid or liquid reagents back into stock bottles. Any excess chemicals can be given to another student or properly discarded under the direction of the instructor.

4. Transfer this sodium chloride to your 100 mL beaker.

5. Transfer a small amount of sand from its reagent bottle to either a weighing cup or to a glass or porcelain container by pouring. Using the milligram balance as you did for the sodium chloride, weigh between 0.300 and 0.400 g of sand and add this to the 100-mL beaker that contains the sodium chloride and mix the contents. Record this mass in your data table. Set the beaker aside until step 9.

6. Tare the balance again and weigh your empty evaporating dish and record the mass in your data table.

7. Obtain a 10-mL volumetric pipet from the back counter of the lab room. Always carry pipets in the vertical position. Obtain a pipet bulb from the counter at the back of the lab room, or drawer 032. Fill a 125-mL Erlenmeyer flask with deionized water, and practice transferring 10.00-mL samples of water to a second 125-mL Erlenmeyer flask using the volumetric pipet. To use a volumetric pipet follow these steps:

   (a) Place the tip of the pipet below the surface of the liquid to be dispensed.

   (b) Compress a pipet bulb and press the opening of the bulb against the upper end of the pipet. *Never force the pipet bulb over the end of the pipet,* it does not fit and it is not supposed to fit.

   **CAUTION:** Always use the pipet bulb, never pipet by mouth.

   (c) Slowly release pressure on the bulb so that liquid is drawn into the pipet to a level about 3 cm above the calibration mark, *but not into the bulb itself.*

   (d) Remove the bulb and simultaneously place your index finger over the end of the pipet. If you are right-handed, you should hold the pipet in your right hand and the pipet bulb in your left.

   (e) Keep your index finger pressed firmly against the end. Withdraw the pipet from the liquid, and slowly reduce the pressure on your finger to allow excess liquid to drain back into its container until the bottom of the meniscus is at the calibration mark.

   (f) Now deliver the remaining liquid into the designated receiver. When releasing liquid from a volumetric pipet, let it drain completely. Wait five seconds, and then touch the pipet tip to the inside of the flask or to the surface of the liquid. This action will remove some, but not all, of the liquid in the tip. The pipet delivers the stated volume when this procedure is followed. There will be a small amount of liquid will remain in the tip. Do not blow this out into your receiver.

 Practice with the pipet until your technique is good. *Before the period ends, you must have the instructor check off your technique for using a pipet.* If this laboratory exercise is being completed virtually, please explain in 4-5 sentences (in the box where the instructor would sign off on your technique) how to properly carry out the volumetric pipetting technique.
8. Pipet two 10.00-mL samples of deionized water into the 100-mL beaker containing the sodium chloride and sand. Stir the mixture in order to dissolve all of the sodium chloride.

9. Attach an iron ring to a ring stand. Obtain a clay triangle from drawer 028 at the back of the lab room, place it on the iron ring, and support a glass funnel on the clay triangle. A clean, dry 100-mL beaker should be placed below the stem of the funnel, with the long end of the funnel’s stem just touching the inside wall of the beaker. This beaker will be used to collect the filtrate.

10. Obtain a circular piece of Whatman Grade No. 4 filter paper (medium porosity, medium flow rate, 8 μm particle retention) from the back counter of the lab room, or from drawer 014. Fold the filter paper along its diameter, and then fold again to form a quadrant.

11. Wet the funnel with deionized water from your plastic wash bottle, then separate the folds of the filter paper, with three thicknesses on one side and one on the other, and place it in the funnel. Wet the filter paper a little more with deionized water from your plastic wash bottle and press the edges firmly against the sides of the funnel so that no air can get between the funnel and the filter paper while the liquid is being filtered.

12. Without disturbing any solid present, carefully pour off the supernatant liquid from your water-sodium chloride-sand mixture into the filter paper. Use a stirring rod to guide the flow. Do not let the supernatant liquid level reach the top of the filter paper, otherwise solid particles may pass through into the filtrate. Rinse the remainder of the solid into the filter paper with a stream of water projected from your deionized water wash bottle. Scrape the beaker with the rubber policeman and rinse the residue it has collected into the filter paper with your wash bottle.
13. To recover the sodium chloride from the filtrate, pour the entire sodium chloride solution from the beaker into an evaporating dish, and then cover the evaporating dish with a watch glass. This should seal the evaporating dish except at the spout.

14. Place the evaporating dish on a ceramic-centered wire gauze, supported by an iron ring on a ring stand. The sodium chloride may be recovered from the solution by boiling off the water. Place a burner below the evaporating dish so that when the burner is lit, the tip of the inner cone just reaches the bottom of the ceramic-centered wire gauze. Light the burner and gently boil heat the solution. When heating solutions, the liquid should boil very gently to prevent the solution from spattering out of the evaporating dish.

**CAUTION:** When heating a liquid to its boiling point, the liquid should come to a gentle boil. If it begins to spatter or boil violently, turn down the flame, or remove the flame momentarily. Do not try to move the liquid container, which will be hot.

Continue to heat the evaporating dish until all of the liquid is gone, and there is no liquid adhering to the inside of the inverted watch glass. Do not overheat, this will cause the salt crystals to spatter out of the evaporating dish.

15. When the water has been evaporated, let the evaporating dish cool, then wash the salt down the sink. Do not run cold water on a hot evaporating dish or it may break.

16. Any collected sand may be placed in the *Sand Recovery Jar*, found in Fume Hood A, and the filter papers may be thrown in the trash can.

17. Dry the outside and inside of the pipet bulb, and return both the dry pipet bulb and the 10-mL volumetric pipet to the back counter of the lab room.

18. Clean and wipe dry your laboratory work area and all apparatus. When you have completed your lab report have the instructor inspect your working area. Once your working area has been checked your lab report can then be turned in to the instructor.
DATA TABLE

| Mass of NaCl | . | g |
| Mass of Sand | . | g |
| Mass of Empty Evaporating Dish | . | g |
| Mass of Evaporating Dish with NaCl After Evaporation | . | g |
| 1 Mass of NaCl Recovered | . | g |
| 2 Percentage of NaCl Recovered | . | % |

Instructor Check for Pipetting Technique

If this is a virtual lab, please write 4-5 sentences on how to properly carry out the pipetting technique using a volumetric pipette. *Do not* just copy the procedure, write it in your own words.

CALCULATIONS

1. 
2. 
POSTLAB QUESTIONS

1. Filter paper should not be wet with water when the liquid to be filtered does not mix with water. Why?
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

2. If the percentage of recovery was less than 100% give some possible reasons why.
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________
_________________________________________________________________________________

3. Do the following metric conversions using dimensional analysis. *Box your answers.*

   (a) 0.185 kilometers to meters

   (b) 74.5 milliliters to liters

   (c) 0.325 kilograms to decigrams