## **Separation Techniques**

**Objective**: The student will learn separation techniques for separating and recovering the components of a mixture of unknown proportions and determine the percentage of each component in the unknown mixture.

**Background**: Mixtures are materials that contain one or more pure substances combined in variable proportions. (Chapter 1, Section 3 of Tro 2<sup>nd</sup> Edition) Mixtures can be classified as homogeneous mixtures or heterogeneous mixtures. A **homogeneous mixture** is a mixture with the same composition throughout. A **heterogeneous mixture** is one whose composition varies from region to region. Mixtures can be separated by exploitation of the individual components differing physical and chemical properties. A binary mixture is a substance that contains only two pure substances.

Basic techniques useful for the separation of mixtures include decantation, distillation, filtration, evaporation, and extraction.

**Decantation** involves separating a liquid from a solid by pouring the liquid, called the **supernate**, from the mixture without disturbing the solid residue.





**Distillation** is a process by in which a homogeneous mixture of liquids can be separated by exploiting their differences in boiling points. Typically the more volatile liquid (lower boiling point) is collected first.

**Filtration** involves pouring a solid-liquid mixture (suspension) through a piece of filter paper or other porous material to separate the solid from the liquid. The solid, called the **residue**, will be left behind in the filter paper while the liquid, called the **filtrate**, will pass through the filter paper.





**Evaporation** is a technique in which the solution is heated and the liquid or **solvent** is vaporized, leaving the solid or **solute** behind.

**Extraction** is the process of selectively solubilizing a component of a mixture by exploiting its unique solubility. A binary mixture of solids can sometimes be separated using the solubility difference of the two solids in water.

#### Safety:

Wear safety goggles. Wash your hands thoroughly before leaving the lab. Do not touch hot objects with your bare skin.

#### Disposal:

Dispose solid residues in the trash.

**Procedure**: All measurements should be recorded using the correct number of significant figures appropriate to the equipment being used. All calculated values should be recorded to the correct number of significant digits. Refer to Chapter 1, Section 7 of Tro 2<sup>nd</sup> Edition about the significant figures.

Preparing Mixture for Separation

- 1. Obtain a vial containing an unknown NaCl-SiO<sub>2</sub> mixture.
- 2. Record the identification code of the mixture on your data sheet.
- 3. Place a clean, dry 100 or 150-mL beaker, labeled beaker "1" on the balance.
- 4. "Tare" or "Zero" the balance.
- 5. Add 2-2.5g of the unknown mixture to beaker "1".
- 6. Record the mass of the unknown solid on your data sheet.
- 7. Add approximately 50 mL of distilled or deionized water to the mixture in beaker 1 and stir the mixture for 2 minutes.
- 8. Obtain a watch glass, ring stand, iron ring, 250-mL beaker, funnel, stirring rod, and a piece of filter paper.
- 9. Place a piece of filter paper on a dry watch glass.
- 10. Weigh the watch glass and filter paper.
- 11. Record this mass on your data sheet.
- 12. Set the watch glass aside for step 29.
- 13. Fold the filter paper first in half and then again in half. Open up the paper cone and place it in the funnel. This folding creates a pocket in the coneshaped paper. The mixture will be filtered through this pocket.
- 14. Label the 250-mL beaker "2"
- 15. Weight the clean, dry 250-mL beaker labeled "2".
- 16. Record this mass on your data sheet.
- 17. Set up your filtration assembly as shown at right using an iron ring to hold the funnel.
- 18. Moisten the filter paper with distilled water from a water bottle so that the paper adheres tightly to the funnel.



- 19. After the solid settles in beaker "1". Pour the liquid into the filter paper. After the majority of the liquid has passed through the funnel, use the stirring rod to guide the solids into the funnel.
- 20. Use distilled water to transfer the remaining solids. (Use water sparingly at this point.)
- 21. Allow all of the liquid to drain into beaker 2 before the proceeding to the next step.
- 22. Place beaker "2" on a hot plate and set the temperature knob to 10. Heat the liquid to boiling. (You can also begin step 29 at this point.)
- 23. Allow the liquid to boil until only a small amount remains in beaker "2".
- 24. Reduce the hot plate setting and continue boiling.
- 25. When the spattering of the solid begins, reduce the hot plate setting to level 1 or 2, and continue heating until all of the solvent has evaporated. (Avoid the spattering of the solid. If the solid spatters at the lowest setting, remove the beaker with beaker tongs until the spattering stops, then place the beaker back on the hot plate.)
- 26. Remove beaker "2" from the hot plate and allow the beaker to cool to room temperature.
- 27. Weigh beaker "2" with the dry solid.
- 28. Record this mass on your data sheet. (Do not discard contents of beaker until you are confident that your solid is dry.)
- 29. Transfer the filter paper and solid to the watch glass massed in step 12 and gently unfold it while held over the watch glass.
- 30. Dry the solid by placing the watch glass and filter paper over a 400-mL beaker of boiling tap water on a hot plate. Continue heating the watch glass and filter paper until the solid is completely dry.
- 31. Remove the watch glass and the solid from the beaker using crucible tongs. Allow the watch glass to cool at room temperature.
- 32. Mass the watch glass, filter paper, and solid.
- 33. Record this mass on your data sheet. (Do not discard the contents of the watch glass until you are confident that your solid is dry.)

# Data

Identification code of unknown mixture	
Mass of mixture, g	
Mass of beaker "2", g	
Mass of beaker "2" and NaCl, g	
Mass of watch glass and filter paper, g	
Mass of watch glass, filter paper, and SiO <sub>2</sub> , g	
Results	
Mass of SiO <sub>2</sub> recovered, g	
Mass of NaCl recovered, g	
Mass of NaCl-SiO <sub>2</sub> recovered, g	
Percentage of original mixture recovered	
w/w % of SiO <sub>2</sub> in sample	
w/w % of NaCl in sample	

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### **Pre-Laboratory Questions**

- 1. Is the sand and salt mixture a homogenous or heterogeneous mixture? Explain.
- 2. Will you be using the extraction technique during this laboratory? If so, describe how you will be using extraction.

3. Will you be using the filtration technique during this laboratory? If so, describe how you will be using filtration, identify the filtrate, and identify the residue.

4. Will you be using the decantation technique during this laboratory? If so, describe how you will be using decantation and identify the supernate.

5. Will you be using the distillation technique during this laboratory? If so, describe how you will be using distillation and identify the component that will be vaporized first.

6. Will you be using the evaporation technique during this laboratory? If so, describe how you will be using evaporation, identify the solute, and the solvent?

### **Post-Laboratory Questions**

- 1. Why is it important to stir the salt, sand, and water mixture for 2 minutes in step 7?
- 2. The percent recovery of your mixture should theoretically be 100%.
  - a. What was your % recovery of the original sample?
  - b. If your % recovery was more or less than the theoretical value of 100%, what is the most likely reason for the error? Explain.

3. What change in the procedure could you make to help ensure a complete recovery?