Resolving a Two-Component Mixture

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Purpose of the Experiment
Separate and recover the components of a mixture of sand and sodium chloride of unknown proportions. Calculate the percent of each component in the mixture and the percent recovery of the components.

Background Required
You should be familiar with basic techniques for measuring volume and mass. You should understand the concepts associated with solution chemistry and stoichiometry.

Background Information
A binary mixture is a mixture containing only two components. We can use a variety of methods, either physical or chemical, to separate the components of such a mixture. Physical separation methods are based on differences between the physical properties of the components, such as solubility or boiling point.

One physical method for separating a liquid from a solid is decantation. First, we allow the solid to settle to the bottom of the container. Then we carefully pour off all of the liquid, called the supernatant liquid, or supernate, into another container, without disturbing the solid.

We can also physically separate a solid from a liquid by filtration, a method that involves pouring the mixture onto a porous material, such as filter paper. The solid, called the residue, is unable to pass through the pores in the filter paper, so it is retained on the paper. The liquid, which passes through the paper, is called the filtrate.

Evaporation is a third physical separation method, in which a solution is heated to vaporize and thus remove the solvent. The solid remaining after we have evaporated the solvent was the solute.

When one component of a binary mixture of two solids is soluble in a particular solvent, and the other component is not, we can separate the components using a fourth physical method, extraction. We add the solvent, dissolving the soluble component, then separate the mixture using filtration.

Example

Determine the percent recovery of silver bromide (AgBr) and potassium bromide (KBr) when separated from a sample of a binary mixture weighing 2.18 g, according to the following procedure.
Water was added to the mixture with stirring in order to extract the KBr. The remaining solid, AgBr, was collected on a piece of filter paper weighing 0.88 g. When dried, the mass of the paper plus the dry AgBr was 1.82 g. The filtrate was collected in a beaker weighing 69.15 g. After evaporation and cooling, the beaker plus the residue weighed 70.33 g.

**Solution**

1. **Calculate the mass of AgBr recovered.**
   
   The mass of AgBr is equal to the mass of the paper plus AgBr minus the mass of the filter paper.
   
   \[
   \text{mass of AgBr, g} = 1.82 \text{ g} - 0.88 \text{ g} = 0.94 \text{ g}
   \]

2. **Calculate the mass of KBr recovered.**

   The mass of KBr is equal to the mass of the beaker plus KBr minus the mass of the beaker.

   \[
   \text{mass of KBr, g} = 70.33 \text{ g} - 69.15 \text{ g} = 1.18 \text{ g}
   \]

3. **Calculate the percent AgBr in the mixture.**

   \[
   \text{percent AgBr in the mixture, } \% = \left( \frac{\text{mass of AgBr recovered, g}}{\text{mass of sample, g}} \right) (100\%) = \left( \frac{0.94 \text{ g}}{2.18 \text{ g sample}} \right) (100\%) = 43\%
   \]

4. **Calculate the percent KBr in the mixture.**

   \[
   \text{percent KBr in the mixture, } \% = \left( \frac{1.18 \text{ g KBr}}{2.18 \text{ g sample}} \right) (100\%) = 54\%
   \]

5. **Calculate your percent recovery of the mixture components.**

   \[
   \text{percent recovery, } \% = \left( \frac{\text{total mass of recovered components, g}}{\text{mass of sample, g}} \right) (100\%)
   \[
   = \left( \frac{0.94 \text{ g} + 1.18 \text{ g}}{2.18 \text{ g}} \right) (100\%) = \left( \frac{2.12 \text{ g}}{2.18 \text{ g}} \right) (100\%) = 97\%
   \]

**In This Experiment**

You will separate and recover sand (silicon dioxide, SiO₂) and table salt (sodium chloride, NaCl) from a binary mixture of unknown proportions, using the procedure outlined in the flowchart in Figure 1 on the next page. After drying and weighing the two recovered compounds, you will calculate the percent of each component in the mixture. You will also calculate your percent recovery of the components.

**Procedure**

**Caution:** Wear departmentally approved safety goggles while doing this experiment. Always use caution in the laboratory. Many chemicals are potentially harmful. Prevent contact with your eyes, skin, and clothing. Avoid ingesting any of the reagents.

**Note:**

- Weigh the portion of mixture you will use for the experiment according to your laboratory instructor's directions.
- Record all masses to the nearest centigram (0.01 g).
- Dispose of your reaction mixtures and rinses according to your laboratory instructor's directions.
- Record all of your data on your Data and Observations sheet.
I. Preparing the Mixture for Separation

1. Obtain a vial containing your unknown NaCl–SiO₂ mixture from your laboratory instructor. Record the identification code of your mixture.

2. Determine the mass of the vial and unknown mixture to the nearest centigram (0.01 g). Record this mass.

   Pour the contents of your vial (2–2.5 g) into a dry 150-mL beaker labeled “1”. Determine the mass of your empty vial. Record this mass.

II. Separating and Recovering the NaCl

A. Extracting the Mixture with Water

3. Measure 50 mL of distilled or deionized water in a graduated cylinder. Slowly add the water to the mixture in beaker 1. Stir the mixture while adding the water and for 2 min afterwards.

B. Preparing the Funnel Assembly for Gravity Filtration

4. Place a funnel in a small iron support ring, as shown in Figure 2.

5. Place a piece of filter paper on a dry watch glass. Weigh the glass and paper. Record this mass. Set the watch glass aside for use in Step 16.

   Note: Do not discard the torn-off corner of your filter paper from Step 6. Place it in your paper cone before beginning the filtration.

6. Fold the filter paper as shown in Figure 3 on the next page. Begin by folding the paper in half (Figure 3b). Make a second fold (Figure 3c), so that the edges of the paper do not quite align (Figure 3d).
Tear off the corner of the smaller section of the paper (Figure 3e). Open up the paper cone and place it in the funnel (Figure 3f). Place the torn-off corner in the paper cone.

7. Label a dry 150-mL beaker “2”. Determine the mass of this beaker. Record this mass.

8. Position beaker 2 under the funnel. Make sure the funnel stem touches the inside wall of the beaker (see Figure 2).

9. Moisten the filter paper with distilled water from a wash bottle. Use a stirring rod to firmly press the wet paper against the funnel walls, so that the paper adheres tightly to the funnel.

C. Filtering the Mixture

10. Decant as much of the supernatant liquid as possible from beaker 1 into the funnel. To prevent splashing and possible loss of solid, use a stirring rod to guide the liquid into the funnel, as shown in Figure 4 on the next page. Collect the filtrate in beaker 2.

   Transfer the solid remaining in beaker 1 into the funnel, using a stream of distilled water from a wash bottle, as shown in Figure 5 on the next page. Use a rubber policeman to transfer any remaining solid into the funnel.

11. After you have transferred all of the solid, rinse the rubber policeman with a stream of distilled water from the wash bottle. Allow the rinses to go into the funnel and drain into beaker 2.

D. Evaporating the Filtrate

**Caution:** In Step 12, as the solution volume decreases, the risk of splattering and sample loss increases. Gentle heating and attentiveness will greatly reduce this risk.

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Figure 3 Folding a piece of filter paper

Tear off the corner of the smaller section of the paper (Figure 3e). Open up the paper cone and place it in the funnel (Figure 3f). Place the torn-off corner in the paper cone.
12. Place beaker 2, containing the filtrate, on a hot plate set on high. Heat the filtrate to boiling.
   Slightly reduce the hot plate setting, and allow the solution to boil gently until only 2–3 mL remain.
   Adjust the hot plate to its lowest setting, and continue heating the solution until all of the solvent has evaporated.

Caution: In Step 13, the beaker will be hot. Handle it with beaker tongs.

13. Turn off the hot plate. Use beaker tongs to remove beaker 2 from the hot plate, as shown in Figure 6.

   Note: You cannot obtain accurate balance readings if you weigh warm objects.

14. Allow beaker 2 to cool to room temperature. Determine the mass of beaker 2 and its contents. Record this mass.
15. Discard the NaCl into the appropriately labeled container.
III. Recovering SiO₂

A. Transferring the SiO₂ Residue to a Watch Glass

16. Carefully remove the filter paper and SiO₂ residue from the funnel. Place the paper and residue on the watch glass you weighed in Step 5.

Use forceps to unfold and open the filter paper.

B. Drying SiO₂

Note: Your laboratory instructor may ask you to store the watch glass, filter paper, and SiO₂ residue in your laboratory bench drawer to dry, and complete the experiment during your next laboratory period.

Caution: In Steps 17 and 18, the hot watch glass and the steam coming from beneath the watch glass can burn your hands.

17. Dry the SiO₂ by placing the watch glass over a 400-mL beaker of boiling water on a hot plate, as shown in Figure 7. Continue heating until the SiO₂ is completely dry.

18. Use crucible tongs to move the hot watch glass and its contents to the laboratory bench. Allow the watch glass to cool to room temperature. Carefully dry the bottom of the glass with an absorbent towel.

Determine the mass of the watch glass, filter paper, and SiO₂ residue. Record this mass.

19. Discard the SiO₂ and filter paper into the appropriately labeled containers.

Caution: Wash your hands thoroughly with soap or detergent before leaving the laboratory.
Post-Laboratory Questions

Use the spaces provided for the answers and additional paper if necessary.

1. Consider your percent recovery for this experiment.
   (a) If you recovered less than 100% of your original mixture, which component do you think was more likely lost during the separation? Briefly explain.

   (b) What procedural change would you make to ensure more complete recovery of the lost component you identified in (a)?

   (c) What procedural error(s) could cause a student to report a percent recovery greater than 100%? Briefly explain.

2. Suggest why the procedure you used specified separation of SiO₂ from the NaCl solution by filtration, rather than by decantation.

3. While performing Step 16 of the Procedure, a student accidentally tore the wet filter paper while opening it. How would his results have been affected?
4. You are given a binary mixture of AgCl and lead chloride, PbCl₂, of unknown proportions. Both compounds are insoluble in cold water, and only PbCl₂ is soluble in hot water.

(a) Devise a method for separating the two components.

(b) Draw a flowchart illustrating your separation scheme.

(c) After completing the separation, you learn that your calculated percent recovery of one of the components is low. For which component was your recovery probably low? Briefly explain.
Data and Observations

- identification code of unknown _______________________________
- mass of vial plus sample, g _______________________________
- mass of empty vial, g _______________________________
- mass of beaker 2, g _______________________________
- mass of beaker 2 plus NaCl, g _______________________________
- mass of watch glass and filter paper, g _______________________________
- mass of watch glass, filter paper, and SiO₂, g _______________________________

Calculations and Conclusions

Show your calculations in the space provided. Remember to include units with all calculated results.

1. Calculate the mass of unknown mixture that you analyzed.
   - mass of unknown _______________________________

2. Calculate the mass of NaCl recovered.
   - mass of NaCl _______________________________

3. Calculate the mass of SiO₂ recovered.
   - mass of SiO₂ _______________________________
4. Calculate the percent NaCl in your unknown mixture.

\[
\text{percent NaCl } \underline{\quad} \quad \underline{\quad}
\]

5. Calculate the percent SiO\(_2\) in your unknown mixture.

\[
\text{percent SiO}_2 \quad \underline{\quad}
\]

6. Calculate the total mass of NaCl and SiO\(_2\) recovered.

\[
\text{total mass recovered } \underline{\quad}
\]

7. Calculate your percent recovery of the components.

\[
\text{percent recovery } \underline{\quad}
\]
Pre-Laboratory Assignment

1. What hazards should you be aware of when evaporating a solvent from a solution?

2. Distinguish between the following pairs of terms, as they apply to this experiment:
   (a) decantation and filtration
   (b) supernate and filtrate
   (c) evaporation and extraction
3. Regarding the techniques you will use in this experiment, briefly explain:
   (a) why you must place the torn-off corner of filter paper into the paper cone (Step 6).
   (b) why you must allow an object to cool to room temperature before you determine its mass.

4. Draw a flowchart for the Example separation in the Background Information.