

Separation of the Components of a Mixture

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Purpose: To become familiar with the methods of separating substances from one another using decantation, extraction, and sublimation techniques.

Equipment: Analytical balance, 2 Bunsen burners, rubber hose, tongs, 2 evaporating dishes, 2 watch glasses, 100-mL graduated cylinder, 2 clay triangles, 2 ring stands, 2 iron rings, glass stirring rods

Materials: Unknown mixture of sodium chloride, ammonium chloride, and silicon dioxide (vial #4)

Introduction: Mixtures are composed of two or more substances mixed together. Mixtures can be homogeneous, or uniformly distributed; they can also be heterogeneous, or not uniformly distributed. The components of a mixture remain chemically unchanged. They are merely physically mixed. Therefore, it is possible to separate them.

There are several ways to separate substances, depending on the properties of the substances. Some substances dissolve when placed in water. These substances are miscible or soluble in water. Others are unchanged when placed in water. These substances are immiscible or insoluble in water. Thus, if a soluble substance is mixed with an insoluble substance, separation through decantation can take place. Decantation involves pouring water onto a mixture and stirring. The soluble substance will dissolve, leaving the insoluble one intact. The newly-formed aqueous mixture of water and the soluble substance can be poured into a separate container and heated so that the water evaporates. The substances have now been separated without any changes to the elemental composition of the substances. Decantation is usually performed more than once on the insoluble substance to ensure that all particles of the soluble substance have been removed.

Some substances can pass directly from the solid to the gaseous stage without first melting and becoming liquid. These substances are said to be able to sublime. Substances that sublime, when mixed with substances that do not sublime, can be separated by heating the mixture until the substance that can sublime is completely gone. One can determine whether sublimation is complete by whether smoke is being produced. If smoke is being produced, then sublimation is occurring.

A third method of separation is called filtration. This is the process of separating a solid from a liquid by means of a porous filter which allows the liquid to pass through but not the solid. This has nothing to do with solubility or ability to sublime, but rather simply the physical phase of the substances and the permeability of the filter.

Antoine Lavoisier's Law of the Conservation of Mass states that in a chemical reaction, matter is neither created nor destroyed. Likewise, in any physical separation, matter is not created or destroyed. The mass of the products of separation should equal the mass of the original substance.

All ionic compounds are soluble in water. Some common substances that can sublime are naphthalene and ammonium chloride. In this experiment, three pre-mixed substances will be separated using the methods outlined above. These substances are sodium chloride (an ionic compound, soluble in water), ammonium chloride (able to sublime), and silicon dioxide (insoluble in water, not able to sublime).

Procedure:

1. A vial of the unknown mixture was obtained. A small, dry evaporating dish was weighed and the mass was recorded. The contents of the vial were emptied onto the evaporating dish. The dish and sample were weighed and the mass was recorded.

2. The dish was placed onto a ring stand. The dish was heated using a Bunsen burner, producing white smoke. When production of smoke stopped, the dish was placed on a cooling pad. Once cool, the dish was weighed and the mass was recorded.

3. A large, dry evaporating dish with a watch glass on it was weighed and the mass was recorded. About 25 milliliters of water was added to the mixture in the small evaporating dish. The contents of the dish were stirred with a glass stirring rod. The water from the small dish was decanted into the larger evaporating dish. Ten milliliters of water was added to the large evaporating dish, and the stirring and decanting was repeated. The decanting was then repeated a third time.

4. Two ring stands were set up. On one ring stand, the contents of the large evaporating dish were heated using a watch glass to prevent spattering and a Bunsen burner. On the other ring stand, the contents of the small evaporating dish were heated using a watch glass to prevent spattering and a Bunsen burner. After having been sufficiently heated to remove water, the dishes were allowed to cool. The dishes were then weighed and their masses were recorded.

Observations: (numbers correspond to procedural steps)

1. Vial obtained: #4

Mass of small evaporating dish: $40.7234 \pm .0001$ g

Mass of small evaporating dish plus sample: $43.7180 \pm .0001$ g

2. Mass of small evaporating dish after subliming NH_4Cl : $43.2652 \pm .0001$ g
(The sublimation process produced white smoke.)

3. Mass of large evaporating dish and watch glass: $120.9955 \pm .0001$ g

4. Mass of large evaporating dish, watch glass, and NaCl : $122.1595 \pm .0001$ g

Mass of small evaporating dish and SiO_2 : $42.0215 \pm .0001$ g

Results:

1. **Mass of original sample.** (Mass of small evaporating dish plus sample *minus* mass of small evaporating dish) =

$$43.7180 \pm .0001 \text{ g} - 40.7234 \pm .0001 \text{ g} =$$

$$2.9946 \pm .0002 \text{ g}$$

2. **Mass of NH₄Cl.** (Mass of small evaporating dish plus sample *minus* mass of small evaporating dish after subliming NH₄Cl) =

$$43.7180 \pm .0001 \text{ g} - 43.2652 \pm .0001 \text{ g} =$$

$$.4528 \pm .0002 \text{ g}$$

3. **Percent NH₄Cl.** (Mass of NH₄Cl *divided by* mass of original sample *times* 100) =

$$.4528 \text{ g} / 2.9946 \text{ g} * 100 =$$

$$15.12\% \text{ NH}_4\text{Cl}$$

4. **Mass of NaCl.** (Mass of large evaporating dish, watch glass, and NaCl *minus* mass of large evaporating dish and watch glass) =

$$122.1595 \pm .0001 \text{ g} - 120.9955 \pm .0001 \text{ g} =$$

$$1.1640 \pm .0002 \text{ g}$$

5. **Percent NaCl.** (Mass of NaCl *divided by* mass of original sample *times* 100) =

$$1.1640 \text{ g} / 2.9946 \text{ g} * 100 =$$

$$38.87\% \text{ NaCl}$$

6. **Mass of SiO₂.** (Mass of small evaporating dish and SiO₂ *minus* mass of small evaporating dish) =

$$42.0215 \pm .0001 \text{ g} - 40.7234 \pm .0001 \text{ g} =$$

$$1.2981 \pm .0002 \text{ g}$$

7. **Percent SiO₂.** (Mass of SiO₂ *divided by* mass of original sample *times* 100) =

$$1.2981 \text{ g} / 2.9946 \text{ g} * 100 =$$

43.35% SiO₂

8. **Total mass recovered.** (Mass of NH₄Cl *plus* mass of NaCl *plus* mass of SiO₂) =

$$.4528 \pm .0002 \text{ g} + 1.1640 \pm .0002 \text{ g} + 1.2981 \pm .0002 \text{ g} =$$

$$2.9149 \pm .0006 \text{ g}$$

9. **Percent recovered.** (Total mass recovered *divided by* mass of original sample *times* 100) =

$$2.9149 \text{ g} / 2.9946 \text{ g} * 100 =$$

97.34% recovery.

10. **Unrecovered mass.** (Mass of original sample *minus* total mass recovered) =

$$2.9946 \pm .0002 \text{ g} - 2.9149 \pm .0006 \text{ g} =$$

$$.0797 \pm .0008 \text{ g}$$

Discussion: The percent recovery for this experiment was low. There were sources of error that affected the amount of substance that could be recovered. During heating, some of the silicon dioxide spattered onto the watch glass. After heating was complete, in an attempt to keep the amount of silicon dioxide consistent, the silicon dioxide on the watch glass was swept into the evaporating dish. However, not all of the substance fell into the evaporating glass. The substance that fell onto the tabletop was not recoverable.

Another source of error is the decanting process. Since this process is subject to human error at every point, it is likely that an error occurred during the process, such as pouring excess water from the original dish into the receptacle dish, causing some of the substance in the first dish to enter the second. However, this is not necessarily a source of

discrepancy in masses, as decanting is a separating process. Separation processes do not affect total mass, but rather individual masses. The individual masses could have been changed by errors in decanting, but not the overall mass.

The theory associated with this experiment is that something must cause the properties exhibited by the substances, such as ability to sublime and solubility in water. These properties are results of the elemental composition of the substances, and more importantly, their structural formulas. Solubility in water depends on whether a substance is ionic or molecular, and whether it will break up in a double replacement reaction when placed in water. Solubility in other substances depends on the structure of both substances. Likewise, the ability to sublime is a result of the structural formula of a substance and its reaction with oxygen gas. All intensive properties like these two are caused by the elemental composition of the substance and the structure of those elements in the substance.

The ramifications for knowing how to separate substances based on solubility are important in industry and in everyday life. Many elements, especially metals, are mass produced by separating them from naturally-occurring substances or ores. Knowing what will cause a separation is essential for causing the separation. Likewise, in everyday applications, knowing how to separate two substances is essential. For example, if while playing in a sandbox, one spilled a sample of lithium chloride into the sand, the sample is not lost. Simply scooping up the sand and the lithium chloride into a beaker would save the sample. To remove the sand from the lithium chloride, one should first check to see whether lithium chloride is soluble in water and whether it will explode in water. Since it does not, and sand is insoluble in water, simply pour water into the mixture, stir, and de-

cant until the sand and the lithium chloride have been separated. Then, boil the lithium chloride and water mixture until all that remains is the original sample of lithium chloride. Next time, simply be more careful with the lithium chloride in the sandbox.

Questions:

1. *Could the separation in this experiment have been done in a different order?* It cannot be done in a different order while still measuring mass. Ammonium chloride is very soluble in water. If the entire original sample was put in water, only the silicon dioxide would be left behind after decantation. Then, when heating the mixture of water, sodium chloride, and ammonium chloride, three things would be happening at once: the evaporation of water, the precipitation of sodium chloride, and the evaporation of aqueous ammonium chloride. Thus, the mass of the ammonium chloride could never be determined. Only the mass of sodium chloride and silicon dioxide could be determined.

2. *How could you separate barium sulfate from ammonium chloride?* Barium sulfate has a very low solubility in water. Ammonium chloride has a very high solubility in water. Thus, the mixture could be put in water and decanted. This would separate the two substances. (source: <http://www.solvayminerals.com/pdf/msds/031.pdf>)

3. *How could you separate zinc chloride from zinc sulfide?* Zinc chloride is very soluble in water. Zinc sulfide is insoluble in water. Thus, the mixture could be put into water and decanted. This would separate the two substances.

(sources: http://www.ilo.org/public/english/protection/safework/cis/products/icsc/dtasht/_icsc10/icsc1064.htm,
<http://www.espimetals.com/msds/s/zincsulfide.pdf>)

4. *How could you separate tellurium dioxide from silicon dioxide?* Tellurium dioxide is insoluble in water. Silicon dioxide is also insoluble in water. The density of tellurium di-

oxide at 293 K is 6.00 g/cm³. The density of silicon dioxide at 293 K is 2.0-2.3 g/cm³. Thus, one theoretical way to separate these materials would be to put the tellurium dioxide at the bottom of a container, put a layer of a liquid with a density between 2.4 g/cm³ and 5.9 g/cm³ and that is insoluble with either solid on top of the tellurium dioxide, and then put the silicon dioxide on top, then carefully decant. This leaves the problem of the new liquid mixed with the silicon dioxide. Separation of those two substance could prove impossible. However, since no liquid has those properties, there is no practical way to separate tellurium dioxide from silicon dioxide. (sources: <http://www.almazoptics.com/homepage/TeO2.htm>, http://www.batnet.com/enigmatics/semiconductor_processing/CVD_Fundamentals/films/SiO2_properties.html)

5. Naphthalene sublimes easily but potassium bromide does not. How could you separate these two substances? Simply put both into an evaporating dish and heat strongly until all of the naphthalene has sublimed. What remains in the dish will be potassium bromide.

Conclusion: The experiment was completed to a high degree of success. Familiarity with the methods of separating substances from one another using decantation, extraction, and sublimation techniques was gained, and the material was recovered to a reasonable degree of accuracy.