SEPARATION OF A MIXTURE OF SUBSTANCES LAB

Purpose: Every chemical has a set of defined physical properties, and when combined they present a unique fingerprint for that chemical. When chemicals are present in a mixture, these unique physical properties can be utilized to separate the chemicals into their pure states. This experiment will give you experience in separating the components in a three-compound mixture; ammonium chloride, sodium chloride (table salt), and silicon dioxide (sand).

Background: Because there are 3 components in our mixture, we will need several techniques to separate them. For this particular exercise, you should be familiar with the following:

- Sublimation: Many chemicals will *sublime* (phase change from solid state to gas state) provided the right conditions. Iodine, for example, sublimes at room temperature. A bottle of iodine will thus always have crystals forming around inside the bottle around the cap where sublimed iodine is being deposited (phase change from gas state back to solid state). Provided the proper equipment, chemicals that are being sublimed can be recovered.
- Decant(ing): *Decanting* is a way of separating a solid from a liquid. We can carefully pour a liquid from a solid, provided the solid is quite dense and resides on the bottom of a container. The easiest way to accomplish this is to hold a glass stir rod against the lip of the container containing the solid and liquid, and gently pour the liquid down the stir rod into a new container.
- Extraction: Extraction is a way to separate two components with different solubilities, that is, *extract* one chemical from another. Extraction relies on a principle called *serial dilution*. Imagine a mixture of small plastic beads and sugar. They can't be separated easily as solids unless you want to physically pick through the particles with a pair of tweezers. However, sugar will dissolve in water and the plastic won't. If we add water to the mixture, we can then pour off the sugar-water from the plastic beads (that is, decanting the solution). However, there will still be some sugar-water left around the beads. We can add more water to the beads, diluting the sugar-water that was left, and decanting again. If we do this several times (three times is typically sufficient) we remove essentially all of the sugar from around the plastic beads. The adding of water multiple times to the plastic beads to remove all of the sugar is serial dilution.
- Evaporation/Boiling: There are many liquids that will *evaporate* quickly at room temperature (nail polish remover and rubbing alcohol, for example), changing phases from liquid to gas. Other liquids evaporate slower at room temperature, and can be heated to boiling to facilitate the evaporation. If we wanted to get the sugar back from our sugar-water in (3) above, we would need to evaporate the water. Sitting at room temperature could take days, but boiling off the water is a much faster way to recover our sugar.
- Taring: We *tare* equipment on a balance to find its mass. If I want to know how much a sample is in a piece of glassware, I must know the mass of the glassware first. For example, I can measure a beaker's mass, then add a sample to it, then remeasure the mass. Subtracting the mass of the beaker from the mass of the (beaker + sample) leaves me with the mass of the sample.

As outlined above, we will be utilizing sublimation, extraction, decanting, and evaporation to separate our components in this exercise. The following table contains relevant physical properties for the chemicals in our mixture:

	Ammonium Chloride	Sand	Salt
State at 25 °C	Solid	Solid	Solid
Melting point	Sublimes at 340 °C	1600 °C	801 °C
Water soluble	Yes	No	Yes

We can easily see from this table that there are clear options on how to separate the chemicals utilizing differences in physical properties. We can dissolve the sample in water to separate the ammonium chloride and salt from the sand (we could *extract* the sample with water, and *decant* off the solution). If we wanted to separate the components in the solution of ammonium chloride and salt, we could; however, we would need to utilize the sublimation point of ammonium chloride, which requires heating the sample to 340 degrees, i.e. we would need a dry sample. Heating the sample to boiling could result in a loss of the ammonium chloride (we know when it gets hot it turns into a gas). With this information at hand, it makes more sense to sublime the ammonium chloride *first*, and then separate the remaining sand and salt using extraction. Then the water from the extraction can be evaporated off. In this way we will have separated our mixture into pure salt, silicon dioxide, and ammonium chloride (although we will not be isolating the ammonium chloride (gas) in this experiment).

Equipment:

- A ring stand with ring and clay triangle, set up in the fume hood
- 2 Evaporating dishes (they look like small, white bowls with a lip on them)
- A watch glass (a curved piece of round glass)
- A stir rod (basically, a glass stick)
- A Bunsen burner
- A 25 mL graduated cylinder

Procedure:

I. Determination of mass of mixture
   a. Tare one of the evaporating dishes on a balance and record the value in space 1 on your data sheet.
   b. Weigh 10 grams of an unknown mixture in the evaporating dish and record the mass in space 2. Make sure you write down which unknown you are using in your experiment.
   c. Record what chemicals are present in the mixture in space 3.

II. Separation of ammonium chloride
   a. Set up the ring stand with a clay triangle and ring in the hood as demonstrated in class; place a Bunsen burner underneath connected to the gas line.

b. Place the evaporating dish with your mixture on the clay ring and turn on the burner. White “smoke” will start to evolve when the sample becomes hot. This is the ammonium chloride subliming into the hood. You will want to stir the sample carefully with a glass stir rod occasionally. Turn the burner off when no more smoke evolves—all of the ammonium chloride is gone from the sample at this point.
c. Let the dish cool naturally on the clay triangle. You don’t want to risk losing your sample and/or breaking glass by picking it up when it is still hot.
d. Measure the mass of the evaporating dish containing the remaining sample and record in space 4. Record the mass of ammonium chloride in the mixture in space 5. Record the chemicals that are remaining in the dish in space 6.

III. Separation of salt

a. Tare the second evaporating dish and watch glass and record masses in spaces 7 and 8. Find the combined mass of the dish and watch glass and record in space 9.
b. Add 25-30 mL of warm water from the tap to the sand—salt mixture, stir for several minutes to dissolve the salt component. Allow the sand to settle. Decant the solution from the first evaporating dish into the second, making sure you don’t transfer any sand. It is better to leave a small amount of solution in the sand than to get the sand in the other evaporating dish (this is because of serial dilution; by the time we add and decant water 3 times, there is hardly any salt left in the first evaporating dish, so if there is some water left, it isn’t going to affect much).
c. Add 5-10 mL water to the first evaporating dish to continue extracting the salt. Decant into the second evaporating dish. Repeat for a total of 3 total extractions.
d. Evaporate the water from the second evaporating dish using a Bunsen burner and ring stand. When the water level gets low, you will want to place a watch glass loosely on top of the evaporating dish so you don’t lose material if it sputters. When you see no vapor evolving, the sample is dry. Turn off the heat and let the dish cool naturally. When it is cool enough to move, record the mass of the evaporating dish and salt in space 10. Find the mass of the salt in space 11.

IV. Separation of sand

a. Place the first evaporating dish on the clay triangle to evaporate any water left in the sand sample. When it is dry, record its mass in space 12. Find the mass of the sand in space 13.

V. Determine the percent composition for each chemical and the overall recovery of your unknown sample using the formula in space 14.

SEPARATION OF A MIXTURE OF SUBSTANCES PRE-LAB DATA SHEET

Name:_______________________________

After reading the introduction to this lab, answer the following questions to prepare you for the experiment. You may want to refer to lecture materials.

1. What technique(s) can be used to remove one solid from a mixture of solids and how?

 ____

 ____

 ____

2. What technique can be used to separate two compounds with different solubilities?

 ____

3. What technique can be used to isolate a solid that is dissolved in a liquid?

 ____

4. What technique can be used to separate a solid from a liquid?

 ____

5. Explain in your own words the principle of serial dilution:

 ____

 ____

 ____

6. What technique removes ammonium chloride from sand?

 ____

7. What is it called when you get the mass of a container on the balance before getting the mass of the container plus a sample? ______________________________
8. What method will you use to isolate the salt from the sand: ________________________
9. What method will you use to isolate the salt from water: __________________________

SEPARATION OF A MIXTURE OF SUBSTANCES LAB DATA SHEET

Name:______________________________________

Partner(s):____________________________________

Unknown number:______________________________

1. Mass of evaporating dish one ______________________g
2. Mass of initial sample+ dish one ______________________g
3. What chemicals are present in this mixture? ______________________

   ____

   ____
4. Mass of evaporating dish after sublimation ______________________g
5. Mass of ammonium chloride sublimed (2-4) ______________________g
6. What chemicals are left in the mixture now? ______________________

   ____
7. Mass of evaporating dish two ______________________g
8. Mass of watch glass ______________________g
9. Mass of evaporating dish two and watch glass (8+7) ______________________g
10. Mass of dish, watch glass, and salt ______________________g
11. Mass of salt extracted (10-9) ______________________g
12. Mass of dried sand and evaporating dish one ______________________g
13. Mass of dried sand (10-1) ______________________g

14. Find the percent composition for all three chemicals.

$$\text{Percent composition} = \frac{mass\ of\ component}{mass\ of\ initial\ mixture} \times 100\%$$

a). Ammonium chloride ______________________

b). Salt ______________________

c). Sand ______________________

15. Total mass recovered (5+9+11) ______________________

Post-lab questions:

16. How could you separate a mixture of Styrofoam beads, sand, and magnesium sulfate (what is in Epsom salt) given the following information:

	Styrofoam beads	Silicon dioxide	Magnesium sulfate
Density	0.20 g/mL	2.2 g/mL	2.7 g/mL
Water soluble	No	No	Yes
State at 25 ^{o}C	Solid	Solid	Solid

____

____

____

____ ______

17. How did your recovered mass compare to the initial mass for this experiment?

____

____

18. What could have caused the mass to be higher than the initial mass?

____

____

19. What could have caused the mass to be lower than the initial mass?

____

____