Experiment 2

FV 8/7/02

USE OF TOP-LOADING AND ANALYTICAL BALANCES: DETERMINATION OF THE WATER OF HYDRATION OF AN IONIC SOLID

MATERIALS: Two porcelain crucibles and covers, clay triangle, tongs, desicooler, Bunsen burner, striker, iron tripod, unknown hydrate.

PURPOSE: The purposes of this experiment are: (1) to compare the precision of an analytical balance to that of a top-loading balance; (2) to determine the percent by mass of water in a hydrate; and (3) to investigate concepts of accuracy and precision, and the mathematical tools used to analyze data.

LEARNING OBJECTIVES: By the end of this experiment, the student should be able to demonstrate the following proficiencies:

1. Identify and use the following equipment: top-loading balance; analytical balance; tongs; crucible and cover; clay triangle; Bunsen burner; desicooler.
2. Explain the function of a desiccant.
3. Explain the difference between mass and weight and know how to measure each.
4. Name ionic solid hydrates.
5. Define precision and accuracy and know the differences between them.
6. Describe the differences between the precision of the analytical and top-loading balances.
7. Explain factors that affect the operation of the balances.
8. Explain why crucibles are heated before an initial mass determination.
9. Calculate the percent by mass of water in an ionic solid hydrate.
10. Describe the relationship between experimental measurements, precision and significant figures.
11. Apply the rules for significant figures.
12. Apply mathematical tools (mean (arithmetic average), deviation from the mean and average deviation from the mean) to analyze data.

DISCUSSION:

A. Top-Loading and Analytical Balances

Principles: Although the terms mass and weight are often used interchangeably, they are different properties. The exact definitions are given in your text.[1] Weight is measured using devices called scales, such as the familiar bathroom scale. Balances are used to determine mass and are available with a variety of maximum capacities and with different precisions, sensitivities and tolerances. The Plebe labs are equipped with two types of balances, top-loading and analytical balances. The top-loading balances in our laboratories are used to determine masses to ±0.01 g while the analytical balances are sensitive to ± 0.0001 g. In general, top-loading balances can measure materials with masses up to several hundred grams whereas the analytical balances are limited to a maximum mass of one hundred grams.

The sensitivity of the laboratory balances makes it necessary to adhere to some strict rules when making measurements. All balances are affected by vibrations of the bench top and by the movement of air currents around the balance pan. These effects increase as the sensitivity of the balance increases. For example, top-loading balances are usually used with the balance pan open to the air. It is necessary to place these balances out of the direct path of room ventilation. They cannot be used in a hood because the drafts are too strong causing the measurements to fluctuate significantly.

Analytical balances can measure the mass of objects to ± 0.0001 g and are even more sensitive to external disturbances than top-loading balances. The balance pan of an analytical balance is housed in a closed chamber with doors. When using an analytical balance to determine mass, it is necessary to close the balance doors to prevent external drafts from moving the balance pan. Even with the doors closed, air movement inside the balance chamber can also cause measurement error. This is usually observed as a wide oscillation of the readout values as the balance

[1]See "Chemistry" by Brown, Lemay, and Bursten, page 14 (footnote) for an explanation.

pan moves in the air currents. As long as the air currents continue, the balance readings will not stabilize. A major cause of air movement inside the balance chamber is placing hot or cold objects on the balance pan, which leads to the creation of convection currents. For this reason, objects should be at room temperature before their masses are measured on an analytical balance.

Another source of error is vibration caused when objects are placed on the table on which the balance is located. Since laboratory balances are very sensitive, they can act as seismometers when books, bottles or elbows are placed on the balance table. In extreme cases, careless action can alter the calibration of a balance resulting in erroneous measurements. For this reason, it is also important that the balances not be moved.

General Precautions when using a top-loading or analytical balance:

1. DO NOT ATTEMPT TO MAKE ADJUSTMENTS TO THE BALANCE YOURSELF. If the balance is not functioning properly, notify your instructor.

2. Be sure the balance pan is clean. If not, remove any solid debris with a soft brush. If a liquid spill occurs, notify the instructor immediately.

3. Do not lean on, rest hands or arms on, or write lab reports on the balance tables.

4. Determine the mass of clean, dry, room-temperature objects only.

5. Clean up after yourself!

6. Close the analytical balance case doors during measurements and when finished.

7. Re-zero the balances between uses and turn off the balances when all work is completed.

B. Precision, Accuracy, Significant Figures and Error Analysis

For a detailed discussion of precision, accuracy, significant figures and error analysis, see Appendices B, E, and J, and the appropriate section of your text [2].

C. Determination of the Water of Hydration of an Ionic Solid

A hydrate is a chemical compound that contains a specific number of moles of water relative to each mole of the primary ionic solid. The number of moles of water present in the hydrate is a definite percent by mass. The water molecules are usually held loosely within the crystal. Moderate heating of the hydrate will drive off the water, leaving the anhydrous, or water-free, ionic solid.

The chemical formula for a hydrate is written in a special form, using a dot between the formulas for the ionic solid and the water. The dot is used to indicate the loose attraction of the water to the ionic solid. The general form for the molecular formula of an ionic solid hydrate is: $MX_a \cdot bH_2O$, where M is a metal cation, X_a is an anion and b is described below.

When naming hydrates, the ionic solid is named following standard rules of nomenclature. The number of water molecules, denoted as b above, is indicated using the Greek prefix (1 = mono; 2 = di; 3 = tri; 4 = tetra; 5 = penta; 6 = hexa; 7 = hepta; 8 = octa; 9 = nona; 10 = deca; 11 = undeca and 12 = dodeca) with the water portion of the compound referred to as "hydrate".

Frequently, when water molecules are removed from the hydrate to yield the anhydrous form of the compound, a color change also occurs. For example, upon heating, dark blue copper(II) sulfate pentahydrate is converted to a bluish white copper(II) sulfate residue.

$$\underset{\text{blue}}{CuSO_4 \cdot 5H_2O\ (s)} \rightarrow \underset{\text{bluish white}}{CuSO_4\ (s)} + 5\ H_2O\ (g)$$

copper(II) sulfate pentahydrate $\rightarrow$ copper(II) sulfate + water vapor

[2] See "Chemistry" by Brown, Lemay, and Bursten, pages 20 - 24.

This color change indicates fundamental changes in the bonding of the molecule. More information on hydrates can be found in your text.[3]

In this experiment, the percent by mass of water in an unknown hydrate will be determined. This is done by heating the sample in a clean, dry crucible to drive off the water of hydration.[4] The crucible and residue are then cooled in a desicooler. The desicooler is a metal container with a tight fitting lid and a small amount of a drying agent or desiccant inside. A desiccant is a compound that rapidly absorbs water from its surroundings. From the masses of the hydrate and anhydrous residue, the amount of water in the sample can be determined. This data can then be used to calculate the percent by mass of water in the hydrate.

PROCEDURE:

Work with a partner to collect data for this experiment.

Part A. Crucible Preparation

1. Determine a method for identifying each crucible so that you can distinguish between Crucible/Cover Set #1 and Set #2. For all of the following steps, make sure the crucible and cover remain as a set. If either the crucible or cover is broken, you must begin the procedure again.

2. The first step in this procedure involves heating the empty crucibles and covers so that they are completely dry and free of all combustible materials such as fingerprint grease. Position a clean crucible and cover on the clay triangle. Place the cover on the crucible so that it is slightly ajar. This will allow any water vapor in the crucible to escape during the heating process.

3. With a Bunsen burner, heat both the crucible and cover for five minutes using a hot flame that has been adjusted so that the tip of the inner cone is just below the crucible. Allow them to cool for one minute on the clay triangle before placing them both in the desicooler using the crucible tongs provided. Do not place hot crucibles on the bench top or on paper. This would cause them to pick up dirt and other contaminants (and possibly ignite the surface).

4. Immediately replace the lid of the desicooler and allow the crucible and cover to cool to room temperature. This will take as long as or longer than the five minute heating period. The dry environment within the covered desicooler will enable the crucible and cover to cool without absorbing water from the air. Keep the lid on the desicooler whenever possible to extend the life of the desiccant.

5. Repeat steps 2-4 with the second crucible and cover. For the rest of the experiment, the crucibles and covers should ONLY be handled with tongs.

6. When the crucibles and covers have cooled to room temperature, place one of the crucibles and its cover on the analytical balance pan and determine their mass. Record this value, including units, in the Data Section.

7. Determine the mass of the same crucible and its cover on the top-loading balance. Record this value, including units, in the Data Section.

---

[3]See "Chemistry" by Brown, Lemay, and Bursten, page 474.

[4]For details on how to heat crucibles, go to http://www.csudh.edu/oliver/demos/heatcruc/heatcruc.htm.

Part B. Unknown Preparation

1. Obtain an unknown hydrate from your instructor. *Record the sample's unknown number in the Data Section.*

2. Place your pre-weighed crucible on a clean sheet of paper and transfer approximately half of the unknown hydrate into the crucible.

3. Determine the mass of the crucible, cover, and unknown, using the same analytical and top-loading balances as used previously. Record the data.

3. Repeat this procedure with the second crucible set using the remainder of the unknown.

Part C. Heating the Hydrate

1. Place Crucible Set #1 on the clay triangle. Adjust the cover so that a portion of the crucible remains uncovered.

2. Heat the crucible, cover, and unknown with a hot flame as described above.

N Answer In-lab Question #1 on page E2-9.

3. After heating the hydrate for 15 minutes, remove the flame and allow the crucible to cool for one minute before placing the crucible and its cover back in the desicooler.

N Answer In-lab Question #2 on page E2-9.

4. Repeat this heating procedure for your second crucible set.

N Answer In-lab Question #3 on page E2-9.

Part D. Mass of the Residue and Clean-up

1. When the crucible set has cooled to room temperature, obtain the mass of the crucible, cover, and residue. Be sure to handle the crucible and cover with tongs. Use the same balances as used previously. Record the data.

2. After obtaining all masses, discard the hydrate residue in the sink and clean out the crucibles using water and a test tube brush.

3. Place the clean crucible sets on a piece of paper towel to drain. Do not place wet crucibles in the desicooler.

4. Return the unknown tube and cork, without washing, to the instructor.

5. Using your data, complete the calculations on page E2-6. Have your instructor check your work, and then enter the values for the mass of the hydrate, mass of water lost, and % by mass of water for Crucible Set #2 in the class results page posted by your instructor. **Note: it is very important that you record the correct number of significant figures for the % by mass of water**. Copy these class results in the Data Section.

Name _____ Section _____

Partner _____ Date _____

DATA SECTION
Experiment 2

Unknown number _____

	Crucible Set #1		**Crucible Set #2**	
	Analytical Balance	Top-loading Balance	Analytical Balance	Top-loading Balance
Mass of crucible and cover				
Mass of crucible, cover and hydrate				
Mass of crucible, cover and residue				
Mass of hydrate				
Mass of water lost				

Summary of Class Results for Crucible Set #2

	Analytical Balance			**Top-Loading Balance**		
Group	Mass of Hydrate	Mass of Water Lost	% by Mass of Water	Mass of Hydrate	Mass of Water Lost	% by Mass of Water
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						

DATA TREATMENT
Experiment 2

For all calculations, report your answers to the correct number of significant figures[5] and the proper units. Show your work in the area provided.

1. Calculate the percent by mass of water in the hydrate for the two determinations using the data from the *top-loading* balance.

Crucible Set #1 _____

Crucible Set #2 _____

2. Calculate the average value for the percent by mass of water in your unknown as determined from the top-loading balance.

Average % Mass _____

3. Calculate the percent by mass of water in the hydrate for the two determinations using the data from the *analytical* balance.

Crucible Set #1 _____

Crucible Set #2 _____

4. Calculate the average value for the percent by mass of water in your unknown as determined from the analytical balance.

Average % Mass _____

[5]See Appendix B - Significant Figures

5. Mean

Using the summary of class results for Crucible Set #2, calculate the class mean for the mass % of water as determined from each of the two balances. (Your answers should have the same number of significant figures as the tabulated values.) These numbers of significant figures indicate the relative precision of the two weighing techniques.

Analytical Balance _____ Top-Loading Balance _____

6. Deviation from the Mean

a. From Appendix J, write the equation used to calculate the deviation from the mean.

b. What does the "deviation from the mean" indicate?

c. Calculate the deviation from the mean for your results from Questions #2 and #4 compared to the class mean. Use the correct number of significant figures.

Analytical Balance _____ Top-Loading Balance _____

7. Average Deviation from the Mean

a. From Appendix J, write the equation used to calculate the average deviation from the mean.

b. What does the “average deviation from the mean” indicate?

c. From the class results for Crucible Set #2, calculate the class average deviation from the mean.

Analytical Balance _____ Top-Loading Balance _____

8. How does the use of a top-loading balance limit the precision of the mass % results? Explain.

Name _____ Section _____

Date _____

N IN-LAB QUESTIONS N

Experiment 2

(Turn in this page before you leave lab.)

1. What is happening to the crucible's contents as you heat it? Do you think the remaining solid in the crucible will have a mass that is greater, smaller, or the same when you weigh it again? Is the solid undergoing a physical or chemical change? Explain. Use complete sentences to express your thoughts.

2. The procedure advises that the cooling of the crucibles to room temperature will take as long as or longer than the heating. Using complete sentences, suggest why this might be so.

3. When an analysis requires decomposition by heating, as the dehydration performed here, the chemist usually reheats the crucible, cover and residue and then determines the mass again to check for a constant mass. Suppose the mass after the second heating is **less** than the mass after the first heating. Using complete sentences, provide a clear explanation for what might cause this observation. Assume that no mistakes were made in carrying out the procedure and that all the equipment was operating properly.