

## SUPPLEMENTARY MATERIAL

(Student Instructions)

### Determination of the Formula of a Hydrate – A Greener Approach

- Objectives**
- To experimentally determine the formula of a hydrate salt.
  - To learn to think in terms of moles.
  - To use an analytical balance.
  - To learn how to name ionic compounds
  - To learn and apply the concepts of green chemistry

**Materials** Unknown hydrate salt, crucibles and covers, crucible tongs, analytical balances, high temperature oven, ring stand, hot plate, wire gauze, beaker of water, stirring rod with rubber policeman.

**Reading** Suggested reading topics: naming ions and ionic compounds, properties of ionic compounds, mole concept, and analytical balance use.

**PreLab Exercises** Answer the following questions.

1. Define “hydrate” and discuss the bonding associated with a hydrate.
2. If a hydrated salt loses 0.3211 g of mass when heated strongly, how many moles of water were driven off? Show your work.
3. Write the chemical formula for Magnesium sulfate heptahydrate.
4. Give the chemical name of the following salt:  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ .

### Introduction

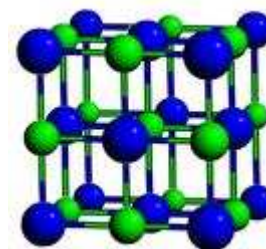
#### *Green Chemistry*

Green chemistry follows a series of 12 Principles (see world-wide web) that seeks to lower the inherent risk of chemical procedures. Green chemistry not only lowers environmental risk, but

also the risk associated with chemical handling. This is achieved by striving to use non-toxic, environmentally benign starting materials, incorporating as much of the starting materials into the desired chemical product, and trying to minimize the amount of waste that is generated in a chemical transformation. One method of minimizing waste is by recycling unwanted products and using them for other types of chemical transformations. In this experiment we will be recycling a dehydrated salt back to its original hydrated form for use in later labs.

### *Hydrates and the mole*

Chemists usually use mole units in describing reactions. The mole is a counting unit, like a dozen, but 1 mole of any substance is  $6.022 \times 10^{23}$  discrete units (instead of 12 in a dozen). This number is known as Avogadro's number. Because the masses of particles (molecules, ions, atoms, etc.) differ greatly, the mass of a mole of particles differs greatly depending on the particle. The mass of a mole of hydrogen atoms is 1.0079 grams, the mass of a mole of potassium chloride salt is 74.5513 grams, and the mass of a mole of insulin molecules is 6,000 grams.



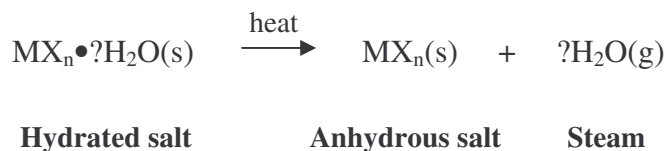
**Crystal Lattice of NaCl**

Salts are interesting in that they can form crystals. The cations and anions orient themselves to allow for the most efficient packing of the ions. This ordered packing is called the *crystal lattice*. Many inorganic salts, when crystallized from water, include water molecules in their crystal lattice. Incorporated water molecules are known as *waters of hydration*. These water molecules are incorporated into the crystal in a definite proportion, but they are not part of the formula of the salt, just a part of the formula of the crystal structure. In other words, copper carbonate with or without water is still copper carbonate. The salt is not defined by the presence of water. By convention, the notation used for these *hydrated* salt crystals takes the form:  $\text{CuCO}_3 \cdot 5\text{H}_2\text{O}$ . This example notates that for

every one  $\text{CuCO}_3$  unit, there are five  $\text{H}_2\text{O}$  molecules in the crystal structure. Similarly, for every one mole of  $\text{CuCO}_3$ , there are five moles of  $\text{H}_2\text{O}$  present. Different salts can have different coefficients of the number of water molecules per formula unit of the salt.

Naming of hydrated salts follows the same method for naming the anhydrous salt (name the cation first, followed by the anion) with the addition of the word 'hydrate' at the end. The number of water molecules is represented by the prefix to the word hydrate. So,  $\text{CuCO}_3 \cdot \text{H}_2\text{O}$  is named copper(II) carbonate monohydrate.  $\text{CuCO}_3 \cdot 2\text{H}_2\text{O}$  is named copper(II) carbonate dihydrate and  $\text{CuCO}_3 \cdot 3\text{H}_2\text{O}$  is the trihydrate salt. The Roman numeral II represents the cation state (charge) of the metal. This annotation is used when more than one charged cation is possible. These multiple charge states are most common for transition metals. For example, copper salts can either be copper(I) or copper(II) salts.

Often, a hydrated salt can be converted into an *anhydrous* salt (one without water) by strong heating to drive off the water molecules. The mass of the hydrate will differ from the mass of the anhydrous salt by the mass of water that was removed during heating. Remember that the masses of the two sides of a balanced chemical equation must be identical due to the conservation of mass. In this way, the number of moles of water originally in the hydrated salt can be determined using the difference between the mass of the hydrated salt and the mass of the anhydrous salt. The “?” is being used since we do not know how many moles of water are being driven off. That is your task for the day!



This reaction can also be reversed to give back the hydrated form by simple exposure of the anhydrous salt to water or water vapor. You will also do this in today's lab. Solids that absorb water on their own from the air are called *hygroscopic* and are useful in creating 'dry' environments.

#### *Green Connections*

Enzymes are important in biology and chemistry since they can catalyze various types of chemical reactions. Enzymes are of particular interest in green chemistry because they are extremely efficient at making the desired product and can often be reused. The waters of hydration of an enzyme play an important role in their function. In some instances, their activity is lost when these waters are removed.

You might be also familiar with hemoglobin. While it is not an enzyme, it serves an important biological function as the oxygen carrier in our blood. Water of hydration is also incorporated into its structure. A recent crystal study of human hemoglobin (PDB structure 1KD2) found the formula  $\text{Fe}_4\text{C}_{2954}\text{H}_{4508}\text{N}_{780}\text{O}_{806}\text{S}_{12}\cdot 243\text{H}_2\text{O}$ . That is 243 water molecules per hemoglobin unit! How's that for

### **Procedure**

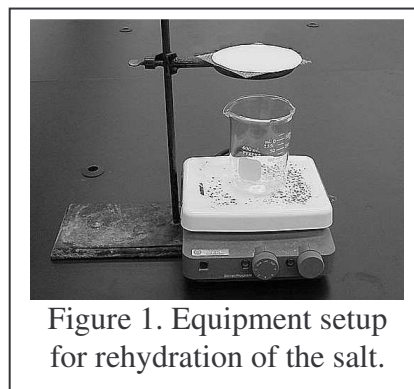
Some items to keep in mind during this experiment:

- Hot glass looks the same as cool glass. Anything that comes out of the oven will be very hot and should not be touched for at least 5 minutes. Remember not to set the hot crucible on the cool bench top since it could break. Place it on wire gauze instead.
- The calculations for determining the formula of the hydrate depend on how accurately you measure the change in mass. Handling the crucible with your fingers may leave behind oils that will change the mass. Also note that the balance will give different readings for the same crucible depending on whether it is hot or cool, so be sure to wait until it is cool.
- It is important that the oven remain at a constant temperature. The only person opening and closing it will be the lab instructor or lab assistant. All of the crucibles will be placed in the oven at the same time.

Prior to lab, the crucibles were placed in the oven to dry. Retrieve a crucible and cover using your crucible tongs, and wait for it to cool. During that time, tare a weighing boat on the top-loading balance and obtain between 0.5 and 0.8 g of the hydrate sample.

Using an analytical balance, record the mass of your empty crucible and cover, transfer the sample from the weighing boat to the crucible, replace the cover, and record the mass again. (Be sure to use the same analytical balance throughout the lab period.) You are now ready to heat your sample. Place the crucible back in the tray and record the position it is in. The crucible will be heated for 30 minutes. During this time you should set up your calculations in your lab notebook.

Once the sample is out of the oven, wait for it to cool and record its mass again. Lift the crucible cover and record your observations. Next you will reheat the sample for 10 minutes to make sure all the water was driven off. During this time you will set up the apparatus for recycling your now dehydrated salt. There are three parts to the set-up: first, set up a hot plate with a 500-mL beaker of water on it. Second, set up a ring stand and attach the ring. On the ring place a piece of wire gauze, and on the wire gauze place a piece of large filter paper. Third, position the stand so that the ring is directly above the beaker of water. When you have completed the set up, turn on the hot plate and bring the water to a boil. Your final setup should look similar to Figure 1.



Once your crucible has come out of the oven a second time, wait for it to cool and record its mass. Is the mass the same as it was after the first heating? Ideally the masses should be within 0.003 g of each other. If there is a large difference, consult with the instructor or lab assistant.

Finally, you need to recycle your sample back into its hydrated form. Remove the wire gauze with the filter paper from the ring stand. Spread the sample out over the filter paper using the stirring rod with a rubber policeman. Carefully place the wire gauze, filter paper, and your sample back on the ring stand.

Mix the sample well as it rehydrates from the steam, but be careful not to push it off the filter paper. You should observe a color change. When the whole sample is completely green-blue again, have it approved by your lab instructor or assistant, and place it in the appropriate container. The next lab section will use your recycled samples, so make sure you are thorough!

### **Analysis and Calculations**

Show calculations to determine:

- the mass of the water driven off
- the number of moles of water driven off
- the number of moles of anhydrous salt (its MW is 134.5 g/mol)
- the formula of the original hydrate (compare numbers of moles of water to number of moles of anhydrous salt). For example:  $\text{MX}_n \cdot ?\text{H}_2\text{O}$ . Give a generic name of your hydrate.

### **Conclusions**

*Provide a summary statement of your work in lab this week. Don't forget to add this experiment to your Table of Contents.*

Answer the following questions:

1. Why must the crucible and lids be heated before the initial weighing?
2. How would your final result (value of “?” in the formula) have been affected if the crucible had been damp before the first weighing? Too high? Too low? Not affected? Explain.

3. Drying agents, or *desiccants*, are sometimes used to keep humidity low inside packaged goods (like leather shoes and purses, or camera equipment). What type of substance encountered in this experiment might serve well as a desiccant? Explain.
4. Unfortunately not all of your original sample will end up in the container for reuse. At what points in the experiment do you “lose” some of the sample? Give an example of one pro and one con of the method you used to recycle the hydrate.
5. This experiment initially used a barium salt to demonstrate the dehydration of a salt. In addition, the salt was thrown away. Propose a reason why you think this does not implement the concepts of green chemistry. Refer to the world wide web for a listing of the 12 Principles of green chemistry.

# SUPPLEMENTARY MATERIAL

(Instructor Notes)

## Determination of the Formula of a Hydrate – A Greener Approach

### General Comments

Traditionally this lab utilizes a Bunsen Burner for salt dehydration, but it has been redesigned to use a 110 °C laboratory oven to carry out the dehydration. Crucibles are oven dried for at least a 30-minute period prior to use during the lab. Samples are simultaneously heated in a covered crucible in a numbered muffin tin for a 30-minute period. Upon initial sample cooling, the students will weigh their crucible and samples and then return their sample to the appropriate muffin cup for an additional 10 minutes to insure complete dehydration. A third heating may be required, but is highly unlikely.

Rehydration of the salt can be carried out with ease. The water does not need to be at a rolling boil for the rehydration to work. The rehydrated salt can be collected in a beaker for reuse.

### Materials

Copper(II) chloride dihydrate (CAS No.: 10125-13-0), crucibles and covers, crucible tongs, analytical balances, high temperature oven, ring stand, hot plate, wire gauze, beaker of water, stirring rod with rubber policeman.

### Hazards

Caution should be taken so that spilling of beakers does not occur during the rehydration process. The copper(II) chloride dihydrate salt should not be sent down the sink or ingested. Filter papers with entrained salt should be disposed of properly.

### Answers to Suggested PreLab Exercises



1. A hydrate is a salt that incorporates a specific pattern of water molecules into its crystal structure. These water molecules are not chemically bound, but are present in a defined stoichiometry with respect to the salt that is present.

$$2. 0.3211 \text{ g } H_2O \times \frac{1 \text{ mol } H_2O}{18.02 \text{ g } H_2O} = 0.01782 \text{ mol } H_2O$$

3. Magnesium sulfate heptahydrate:  $MgSO_4 \cdot 7H_2O$

4.  $FeCl_3 \cdot 6H_2O$  = Iron(III) chloride hexahydrate

### Data and Measurements

Units should be apparent on measurements and significant digits should be sensible given the balance used (analytical balances give 4 decimal places while top-loading balances give two decimal places). A data table should be present and easy to follow.

### Observations

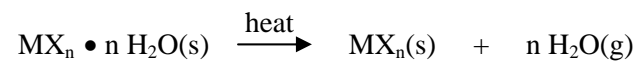
Students should notice the colors changes occurring during the dehydration and later rehydration upon exposure to steam.

### Analysis and Calculations

- Mass of water driven off:  
 $(\text{mass hydrate} + \text{crucible}) - (\text{mass anhydrous salt} + \text{crucible}) = \text{mass water}$
- Moles water driven off:  $\text{mass water (from above)} / \text{FW water} = \text{moles water}$
- Moles anhydrous salt:  $\text{mass anhydrous salt} + \text{crucible} - \text{mass crucible} = \text{mass anhydrous salt}$  then divide by 134.5 g/mol to get moles anhydrous salt.
- Value of ? should be = 2.  $(\text{mass anhydrous salt} / 134.5)$  gives moles of anhydrous salt. Moles of water driven off / moles of anhydrous salt = moles of water given off per moles of salt.

## Answers to Conclusion Questions

1. To make sure it is excruciatingly dry before the hydrate is added for its original weighing.
2. The original crucible mass would have been too high. This moisture would have been removed by heating, just as the hydrated waters were removed. So, more water would have been lost. We would have thought that more water was associated with the salt than there really was.
3. The anhydrous salt would be a good desiccant—it can absorb moisture from the atmosphere to become the more stable hydrate. (If the hydrate was not more stable it wouldn't form spontaneously.)
4. One advantage of using the steam method for rehydration is the speed. The salt could be left on the bench top to rehydrate, but it would take much longer. A disadvantage of using this method is that some salt becomes entrained in the filter paper and can't be recovered.
5. First of all, barium is highly toxic! Green chemistry emphasizes the use of non-toxic substances. Another principle of green chemistry is the recycling and reuse of materials for future reactions. Just throwing the salt away creates waste! By rehydrating it, the salt can be used over and over again. This recycling also helps decrease cost.



Scheme 1. Dehydration of a metal hydrate salt.