

EXPERIMENT 5: HYDRATES

Introduction: You will determine the percentage of water in a hydrate.

Background: Hydrates are chemical compounds that contain water as part of their crystal structure. This water is strongly bonded, is present in a definite proportion, and is referred to as water of hydration or water of crystallization. The formula of a hydrate consists of the formula of the anhydrous (without water) compound followed by a dot, then the number of molecules of water that crystallize with one formula unit of the compound, then the formula of water.

Examples of hydrates are

$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	Calcium sulfate dihydrate
$\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	Cobalt (II) chloride hexahydrate
$\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$	Sodium carbonate monohydrate

Hydrates can be converted to the anhydrous form by heating:



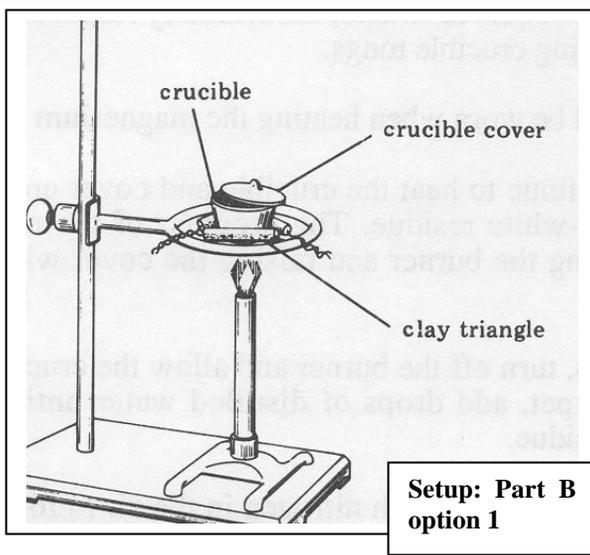
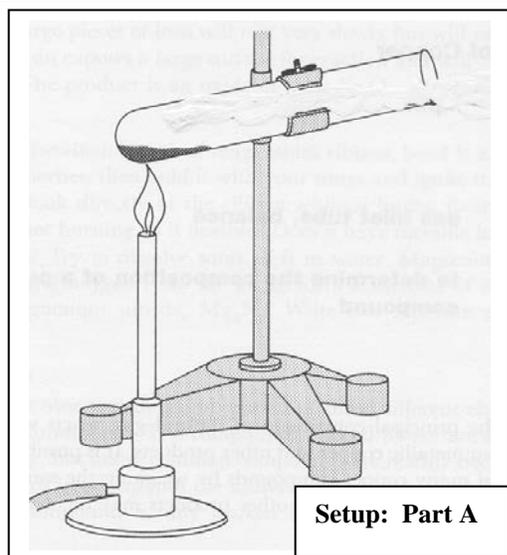
Therefore, you can determine the percentage of water in a hydrate by determining the mass lost (amount of water driven off) when a known mass of hydrate is heated.

$$\text{Percentage of water} = \frac{\text{Mass of water lost}}{\text{Mass of hydrate}} \times 100$$

In this experiment you will “heat to constant mass” in order to make sure that all the water of hydration is driven off. Since you cannot tell if all of the water has been driven off after the first heating or if some water still remains, the procedure of heating and cooling and weighing is repeated until two consecutive weighings are the same, within the limits of the uncertainty of the balance (± 0.02 g).

Materials Needed

Equipment	Chemicals
Pyrex test tube, holder, iron ring, triangle, tongs, Crucible and cover or evaporating dish, cooling pad	$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, Unknown hydrate



Procedure

Part A: The behavior of a hydrate

1. Place a scoop of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (copper (II) sulfate pentahydrate) in a **Pyrex** test tube. There is no need to weigh the sample, as this is a qualitative test. Hold the test tube in a clamp, tilted upward at about a shallow angle, with its mouth pointed away from you and all others (see diagram pg 31). Heat the bottom of the test tube gently. Observe and record all changes seen.
2. **After the test tube has cooled to room temperature**, add a few drops of water. Touch the bottom of the test tube carefully. Record your observations.

Part B: Determining the percentage of water in a hydrate

1. Obtain a sample of an unknown hydrate as directed by your instructor.
2. Weigh a clean dry crucible and cover or an evaporating dish to 2 decimal places. Your instructor will inform you of which container you should use.
3. Place about 1.5 g of the unknown hydrate in an evaporating dish or a crucible and replace the cover, then weigh the container and contents to 2 decimal places.
4. Place the container on a clay triangle. If you are using the cover, have the cover slightly ajar, so water vapor can escape. Heat **VERY GENTLY** for about 5 minutes. Then, immediately adjust the flame so the tip of the inner blue flame just touches the bottom of the crucible, and heat 10 to 15 minutes. The crucible bottom and/or the clay triangle should glow with a brick-red color during this heating period.
5. Turn off the burner, and cool for at least 10 to 15 minutes. Weigh the container after it has completely cooled to room temperature.
6. Heat the container again for about 10 to 15. Cool and weigh.
7. If all the water of hydration was driven off during the first heating, the weights after the first and second heatings should be the same, ± 0.02 g. If the change in mass between the two weighings is more than 0.02 g, repeat the process of heating and cooling and weighing until two successive weighings agree within ± 0.02 g.
8. Calculate the percentage of water in your sample.
9. Calculate the empirical formula of your hydrate.

Safety and Waste Disposal

Safety: Blue burner flames may be hard to see; be careful not to reach across a flame. Long hair should be tied back. Handle hot crucibles or lids only with crucible tongs. The iron ring and clay triangle get very hot, and cool slowly. **GOGGLES MUST BE WORN.**

Never set a hot crucible on the counter. Use a cooling pad or your wire gauze

Waste Disposal: Excess or spilled hydrate or anhydride should be placed in labeled waste containers in the hood.

Part B: Quantitative determination of percentage of water in an unknown hydrate

Data: Unknown # = _____

Mass of Crucible and Cover	
Mass of Crucible, Cover, and Hydrate	
Mass of Crucible, Cover, and Sample After first heating	
Mass of Crucible, Cover and Sample After second heating	
Mass of Crucible, Cover and Sample After third heating, if necessary	

Calculations: Show setups, with numerical values and units. Give answers to the correct number of significant figures.

Calculate the mass of the hydrate used.	
Calculate the mass of water lost by heating.	
Calculate the percentage of water in the unknown hydrate.	

Calculation of Empirical Formula.

Background: All ionic substances are represented by formulas which show the **simplest** whole number ratio between the elements. The simplest formula is also called the **empirical** formula. Given the percent composition of the compound, an empirical formula can be calculated. In this experiment, we treat the water molecules as single units rather than breaking them down into H and O

Example: Given the percent composition of hydrated zinc sulfate, as follows: % (per 100 g) can be converted to mole ratios and then to **simplest mole ratio**.

$$\text{Zn SO}_4 \dots\dots\dots 56.15\% / 161.45 = 0.3477 \text{ mole} \quad 0.3477 / 0.3477 = 1$$

(divide % by molar mass = 161.45)

$$\text{H}_2\text{O} \dots\dots\dots 43.85\% / 18.01 = 2.435 \text{ mole} \quad 2.435 / 0.3477 = 7$$

(divide % by molar mass = 18.01)

Thus the formula of the hydrate is $\text{ZnSO}_4 \cdot 7 \text{H}_2\text{O}$

Experimental values for unknown # _____ (refer to previous page)

% water _____ % anhydride _____ Show these to the instructor.
(experimental value) (100 - % water)

The instructor will now give you the formula of the anhydrous portion of the salt.

The % water in sample: _____ (from instructor)

Percent error for water: _____

Formula of anhydride: _____ (from instructor)

The complete empirical formula of the hydrated salt can now be calculated (show set-up below **similar to** the ZnSO_4 example above). Your sample is probably **not** zinc sulfate, but some other compound.

Formula of the hydrated substance: _____

QUESTIONS AND PROBLEMS

1. How do you know that all of the water of hydration has been removed from your unknown hydrate? Explain.
2. 4.817 grams of an unknown hydrate are heated until all the water is removed. The anhydride residue weighs 4.301 grams. Calculate the percentage of water in this hydrate.
3. a) A student records the following data in the laboratory when determining the percentage water in an unknown hydrate. What is the percentage water in this student's unknown?

Mass of container	78.311 g
Mass of container and hydrate	81.055 g
Mass of container and contents after:	
First heating	80.899 g
Second heating	80.137 g
Third heating	79.999 g

- b) Based on the values obtained in the three heatings above, do you think the student should have confidence in his results? Why? What do you think the student should do to be more confident in his result?