# EXPERIMENT 7: HYDRATES

**Introduction:** You will determine the percentage of water in a hydrate and the empirical formula of a hydrated salt.

**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

| | |
|---|---|
| $CaSO_4 \cdot 2H_2O$ | Calcium sulfate dihydrate |
| $CoCl_2 \cdot 6H_2O$ | Cobalt (II) chloride hexahydrate |
| $Na_2CO_3 \cdot H_2O$ | Sodium carbonate monohydrate |

Hydrates can be converted to the anhydrous form by heating:

Hydrate $\rightarrow$ Anhydride + Water

$$CaSO_4 \cdot 2H_2O \rightarrow CaSO_4(s) + 2\,H_2O(g)$$

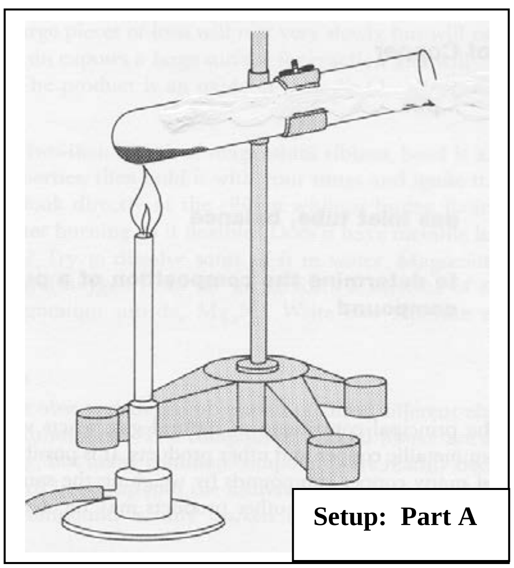
Therefore, you can determine the percentage of water in a hydrate by determining the mass lost (amount of water driven off) when a known mass of hydrate is heated.

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

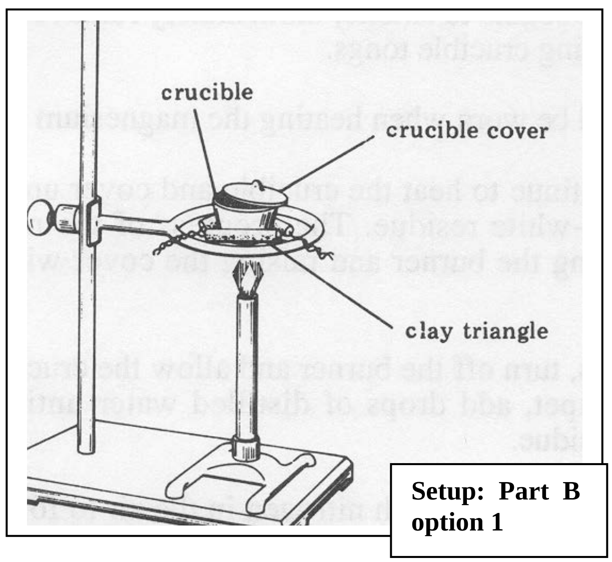
In this experiment you will “heat to constant mass” in order to make sure that all the water of hydration is driven off. Since you cannot tell if all of the water has been driven off after the first heating or if some water still remains, the procedure of heating and cooling and weighing is repeated 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 | $CuSO_4 \cdot 5H_2O$, Unknown hydrate |



**Setup: Part A**




**Setup: Part B option 1**

## Procedure

### Part A: The behavior of a hydrate

Place a scoop of $CuSO_4 \cdot 5H_2O$ (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.

**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

Obtain a sample of an unknown hydrate as directed by your instructor. Either weigh a clean dry crucible and cover or an evaporating dish to 2 decimal places or a clean evaporating dish. Your instructor will inform you of which container you should use.

Place about 1.5 g of the unknown hydrate in an evaporating dish or in a crucible with its cover, then weigh the container and contents to 2 decimal places. If you are using the evaporating dish, place it on wire gauze but if you are using the crucible, place the crucible 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.

Turn off the burner, and cool for at least 10 to 15 minutes. Weigh the container after it has completely cooled to room temperature. Heat the container again for about 10 to 15. Cool and weigh. 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. Calculate the percentage of water in your sample. 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.

**EXPERIMENT 7: REPORT** **Name ____________________**
**HYDRATES**

**Section ______________**

**DATA AND CALCULATIONS:**
**Part A: The behavior of a hydrate**

1. Describe the appearance of the copper(II) sulfate pentahydrate, $CuSO_4 \bullet H_2O$, before heating.

2. Describe the appearance of the anhydride obtained by heating the hydrate. (The anhydride is the residue.)

3. Describe the appearance of any other substances seen during heating, other than the hydrate and anhydride.

4. Describe all changes observed when a few drops of water were added to the anhydride.

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

**Data:** Unknown # = ______________

Weight of container and sample before heating _______________g

Weight of container (either evaporating dish or crucible and cover) _______________g

Weight of sample _______________g

Weight of container and sample after first heating _______________g

Weight of container and sample after heating to constant weight _______________g

Weight of water in sample _______________g

Percent of water in sample ________________%

Calculations:

Correct percent of water in sample (from instructor).. . . . . . . . . . . . . _________________%

Percent error . . . . . . . . . . . .. . . . . . . . . . . . . . . . . . . . . . . . . . . . . . .... . _________________%

Calculations:

Question: How do you know that all of the water of hydration has been removed from your unknown hydrate? Explain.

**Part C: Quantitative determination of empirical formula.**

**Background:** All ionic substances are represented by formulas which show the s**implest** 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.**

Zn $SO_4$…………. **56.15**% / 161.45 = 0.3477 mole 0.3477 / 0.3477 = **1**
(divide % by molar mass = 161.45)

$H_2O$ ……………. **43.85%** / 18.01 = 2.435 mole 2.435 / 0.3477 = **7**
(divide % by molar mass = 18.01)

Thus the formula of the hydrate is $ZnSO_4 \cdot$**7** $H_2O$

**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.

Formula of anhydride: ________________________ (from instructor)

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

## QUESTIONS AND PROBLEMS

1. 2.362 grams of an unknown hydrate are heated until all the water is removed. The anhydride residue weighs 2.015 grams. Calculate the percentage of water in this hydrate.

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2a) 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 | | 47.952 g |
| Mass of container and hydrate | | 49.837 g |
| Mass of container and contents after: | | |
| | First heating | 49.500 g |
| | Second heating | 48.918 g |
| | Third heating | 48.811 g |

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2b) Based on the values obtained in the three heating's above, do you think the student should have confidence in his results? Why? What do you think he should do to be more confident in his result?

3. 10.00 grams of a sample of hydrated $PtCl_4$ are heated and lose 3.00 grams of water. How many moles of water are combined with each mole of $PtCl_4$?

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4. Analysis of a sample of hydrated salt shows that it contains 4.86g Mg, 6.20g P, 11.20g O, and 5.40g $H_2O$. What is the formula of the hydrated salt?

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