

## Determination of Water Content of an Ionic Hydrate

### Introduction

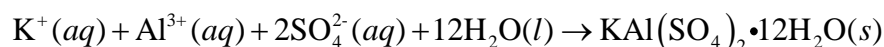
Water of crystallization or water of hydration are water molecules crystallized together with an ionic salt. The salts with such water are called hydrates.

The molecular formula of hydrates consists of the molecular formula of the salt followed by a raised dot, the number of water molecules and "H<sub>2</sub>O" per unit salt. In the names of hydrates the number of water molecules of water per unit salt is given with Greek prefixes and the term "hydrate".

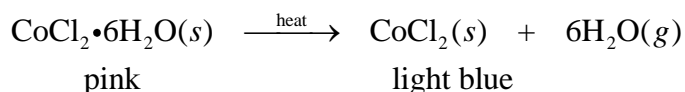
For example

- Cobalt(II)chloride hexahydrate: CoCl<sub>2</sub> • 6H<sub>2</sub>O
- Sodium sulfate decahydrate: Na<sub>2</sub>SO<sub>4</sub> • 10H<sub>2</sub>O

Water of hydration is not the same as a substance being wet. Water molecules in hydrates are incorporated in the crystal or attached to the central ion in well-defined stoichiometric ratios. Hydrates crystallize from aqueous solutions. An example is alum, which you will synthesize later this semester:



The water can be removed by heating, and the left-over salt is the anhydrous salt. For example:

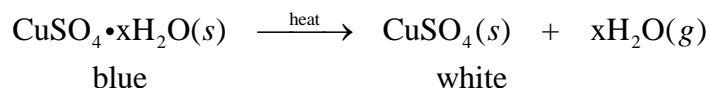


Hydrates of transition metals are usually colorful. Usually the anhydrous version of these salts are hygroscopic, that is they tend to regain the lost water of hydration, which is why some of them are used as desiccants.

Consider a sample of BaCl<sub>2</sub>•xH<sub>2</sub>O heated in a crucible. The mass of the empty crucible was 25.4583 g, the mass of the crucible with a sample was 26.5908 g before, and 26.4239 g.

Mass of the sample before heating: (mass of crucible + sample) - (mass of empty crucible)	26.5908g – 25.4583g = 1.1325 g
Mass of the sample after heating: (mass of crucible + anhydrous salt) - (mass of empty crucible)	26.4239 g – 25.4583 g = 0.9656 g
Mass of water released: (mass of sample) – (mass of anhydrous salt)	1.1325 g – 0.9656 g = 0.1669 g
Amount of water released: (mass of water)/(molar mass of water)	$\frac{0.1669\text{g}}{18.00 \frac{\text{g}}{\text{mol}}} = 9.272 \times 10^{-3} \text{ mol}$
Amount of anhydrous salt formed: (mass of anhydrous salt)/(molar mass of anhydrous salt)	$\frac{0.9656\text{g}}{208.2 \frac{\text{g}}{\text{mol}}} = 4.638 \times 10^{-3} \text{ mol}$
Molar ratio of H <sub>2</sub> O/BaCl <sub>2</sub> : (amount of water)/(amount of anhydrous salt)	$\frac{9.300 \times 10^{-3} \text{ mol}}{4.638 \times 10^{-3} \text{ mol}} = 1.999 \approx 2$
Molecular formula of sample:	BaCl <sub>2</sub> •2H <sub>2</sub> O

In this experiment you will determine the number of water molecules in the hydrate of copper sulfate,  $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$  by heating a sample of the hydrate until it loses all the water of hydration:



### Procedure

1. Weigh a crucible with a lid and record the mass to the nearest 0.0001g.
2. Place the crucible with a lid on a clay triangle mounted on a lab stand (see Figure 1.).
3. Light the Bunsen burner. Heat the crucible until the bottom of the crucible is glowing. Remove the lid periodically with tongs to check the crucible. Place the crucible and the lid on the wire gauze.
4. Allow the crucible and the lid to cool for five min, and then measure its mass. Record the mass.  
**Note:** Make sure that the crucible and the lid are not hot anymore before placing them on the balance.
5. Repeat steps 2-4 again until two consecutive measurements are within 0.001g.
6. Transfer about 1.0 g ( $\pm 0.1$  g) unknown into the cooled down crucible, weigh it with the lid, and record the mass.
7. Start to heat the crucible *gently* by moving the flame around the bottom of the crucible (see Figure 2.).

**Note:** If the heating is too fast, the substance will spatter out from the crucible. Fast heating can turn the sample brown, which is a sign of chemical decomposition of the sample. In which case the experiment has to be repeated from the beginning.

8. Lift the lid periodically and check the contents. When the crystals on the surface turn white, use a straightened paper clip to mix the contents of the crucible (see Figure 3.) allowing the remaining blue crystals to get in contact with the hot wall of the crucible. Once the contents are mixed, replace the lid.
9. When the entire contents turn white (a slightly greenish tint might be present), stop the heating, and move the crucible and the lid to a wire gauze to allow it to cool for 5 min.
10. Weigh the crucible and the lid after you make sure that they cooled down.
11. Dispose of the residue from the crucible as instructed, clean the crucible and the lid.

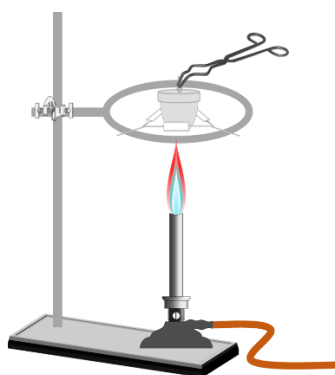


Figure 1.

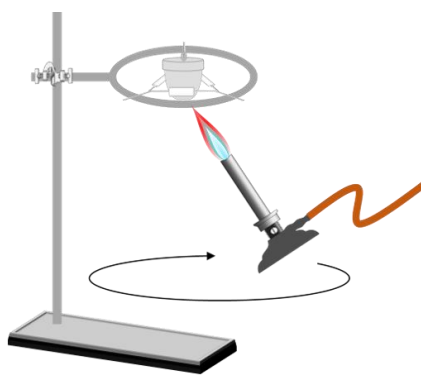


Figure 2.

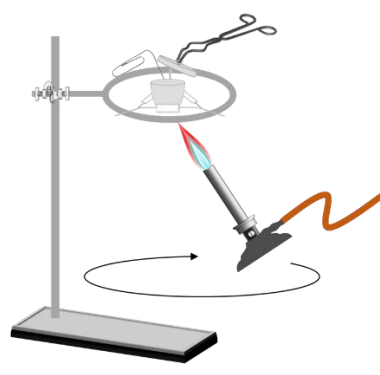


Figure 3.

### Data Table

Mass of empty crucible + lid	After heating 1:	
	After heating 2:	
	After heating 3:	
	After heating 4:	
	After heating 5:	
	Final mass:	
Mass of crucible + lid+ sample:		
Mass of crucible + lid+ sample after heating:		

### Calculations

Mass of original sample:

Mass of sample after heating:

Mass of released water:

Number of moles of the anhydrous salt:

Number of moles of water released:

Molar ratio between water and anhydrous salt ( $\text{H}_2\text{O}/\text{CuSO}_4$ ):

Molecular formula of hydrated salt:

**Note:** Attach or reproduce your Data Table and Calculations in your Report. If you attach them, reference them in your Report.



## Pre-Lab Questions

1. Explain the difference between the water of hydration and a sample being wet.
2. Do you include the water of hydration when you calculate the molar mass of a hydrate?
3. Desiccants are very hygroscopic materials used to dry wet samples. When they are spent and can no longer absorb moisture they need to be replaced. Knowing that the anhydrous  $\text{CoCl}_2$  is blue, and the hydrate,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ , is pink, why do you think the anhydrous  $\text{CoCl}_2$  can be used as an "indicator" in desiccants to show when they need to be replaced?
4. Would you consider the losing of water of hydration a physical, a chemical process, or both? Explain.
5. In the procedure, why do you think you may have to heat the crucible initially, maybe more than once?



## Post-Lab Questions

1. Why do you think you had to keep the lid in the measurements?
2. Why do you think your molar ratio of water and  $\text{CuSO}_4$  was not an integer?
3. Why do you need to round the experimental ratio of water and  $\text{CuSO}_4$  to the nearest integer?
4. How would your result be affected if some blue crystals were left unchanged? Explain.
5. How do you think your results would be affected if the sample is overheated and some of the  $\text{CuSO}_4$  decomposed to  $\text{CuO}$ ?