

Objectives

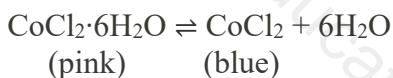
- Calculate the percent water by mass in several hydrated compounds
- Dehydrate an unknown solid sample and identify it by comparing its percent water with known hydrated compounds

Background

Waters of Hydration

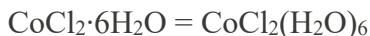
Many solids, especially inorganic salts, occur naturally as hydrates. This means that water molecules are incorporated into the crystal lattice of the compound in such a way that the water is chemically bound. Some of these materials may spontaneously lose water molecules when placed in contact with dry air, a process known as efflorescence. Other hydrates are hygroscopic, meaning that they adsorb water from humid air. These materials are often used as desiccants, or drying agents. For example, many electronic products are shipped with small packets of silica which acts as a desiccant to protect the electronics from the effects of humidity. Some solids absorb so much humidity from the air that they then start to dissolve in the large quantity of absorbed water, a process known as deliquescence.

Some hydrates exist in equilibrium with moisture in the air, absorbing or releasing water molecules depending on the relative humidity. One example is cobalt (II) chloride, which adsorbs or releases water in a reversible process:



Cobalt chloride is used in many novelty items, such as weather strips to predict the weather. When the chance of rain increases and the relative humidity is high, the cobalt chloride absorbs water to form the hydrated salt and the weather strip turns pink. When the relative humidity drops during fair weather the compound releases water and reverts to the anhydrous form, and the weather strip turns blue.

Hydrated salts are not simply solids that are wet or that have moisture adsorbed on their surface. They are compounds in which water molecules are incorporated into the crystal structure of the salt, chemically combined with the cations and anions. This is reflected in the chemical formula of the salt, which includes the specific number of water molecules in the unit cell (or moles of water per mole of salt). Thus, the hydrated cobalt chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) includes 6 water molecules in its unit cell, or 6 moles of water per mole of salt. The common notation for hydrated salts is to use a raised dot (\cdot) between the formula for the salt and the number of water molecules in the compound. Alternatively, the formula for hydrated salts can be written with the water molecules in parentheses, with the number of water molecules indicated using a subscript. In other words:



Because water is part of the chemical structure of a hydrated salt it is important to specify the amount of water when naming hydrates. The rule for naming hydrated salts is to start with the name of the salt, and add the term “hydrate” with a Greek numerical prefix to indicate the molar ratio of water in the compound. These prefixes are listed in Table 1.

Table 1 Greek numerical prefixes

Number of water molecules	Prefix
1	mono
2	di
3	tri
4	tetra
5	penta
6	hexa
7	hepta

Thus, the name of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ is “copper (II) sulfate pentahydrate”. The name of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ is “barium chloride dihydrate”.

In this exercise, you will identify an unknown hydrated salt from a list of potential unknowns. Water of hydration is easily removed from the compound by heating it to above 100oC (the boiling point of water). By measuring the mass of water lost upon heating from a given amount of hydrated salt, the % water by mass can be calculated and compared to the theoretical values based on the formulas of known compounds.

Materials

- Hydrated salts
 - $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$
 - $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$
 - $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$
 - $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$
 - $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$
- Wire triangle and iron ring
- Crucible with lid
- Crucible tongs
- Bunsen burner

Safety goggles are required!

Handle the crucible, lid, wire triangle, and iron ring carefully—objects remain hot long after heating is discontinued. Solid samples may tend to spatter during heating.

Procedure

Waters of Hydration

1. Clean a porcelain crucible and cover. Do not worry about stains that will not come off. Support the crucible and lid (slightly ajar) on a clay triangle on a ring stand. Heat gently on a hot plate for a minute, and then heat strongly to redness for about three minutes. Allow to cool completely. Allow to cool *completely*.
2. Using crucible tongs or a paper towel, transfer the crucible and lid to an analytical balance and weigh them to the nearest 0.001 g. Never touch the crucible or cover with your bare hands as oils and moisture from your skin will add mass to the crucible; always use crucible tongs to handle the crucible.
3. Select an unknown and record the ID code on your Data Sheet. Transfer about 1 gram of unknown to the crucible and reweigh to the nearest 0.001 g. The mass of the unknown sample can be determined by difference.
4. Set the crucible and cover over the Bunsen burner. The lid should be slightly ajar to allow steam to escape during heating. Heat gently for about 5 minutes—vigorous heating of the sample may cause it to splatter or to undergo decomposition. After several minutes, lift the lid to observe any changes in the material. When heating is finished, replace the lid to completely cover the sample to prevent re-adsorption of water, and allow the crucible to cool.
5. When the crucible and lid have cooled completely, weigh the crucible and *anhydrous* salt sample. Record the mass to the nearest 0.001 g on your data sheet.
6. Return the crucible and lid to the clay triangle and heat gently for another 5 minutes. Cool completely and weigh, recording the mass on your data sheet. If the weight after the second heating agrees with the mass after the first heating (within 0.005 g) then no further heating is necessary. If the second heating appeared to drive off additional water then heat a third time, cool, and re-weigh. Continue until you obtain agreement between successive weighings.

- Scrape out the anhydrous salt into an evaporating dish or small beaker. Record any observations regarding the appearance of the anhydrous salt.
- Repeat steps 2–6 using a second sample of the same unknown. Calculate the % water by mass in each sample. Average your values for the two trials.
- Add the anhydrous salt from the second trial to the evaporating dish or beaker, and add a few drops of water. Record any changes in the appearance of the salt on your data sheet. Dissolve any remaining solid and rinse down the sink with plenty of water. Clean the crucible and cover, and return to the lab cart or lab drawer as instructed.

Calculations

Percent Water by Mass

- Calculate the mass of each hydrated salt sample by difference.
- Calculate the mass of water lost as the difference between the mass of the hydrated salt and the mass of the anhydrous salt.
- Calculate % water by mass in each sample as:

$$\% \text{ water} = \frac{\text{mass of water lost}}{\text{mass of hydrated salt}} \times 100 \quad \text{Equation 1}$$

Record the % water by mass for each trial on your data sheet. Calculate the average % water for your trials.

Unknown Identification

- Calculate the theoretical % water by mass for each of the possible unknowns listed on your data sheet. The simplest way to calculate the mass % of water is to begin by assuming one mole of the compound. The % water by mass can then be determined as the mass of water divided by the mass of compound, times 100%.

For example, the % water by mass in $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ would be calculated as follows:

$$\% \text{ water} = \frac{\text{mass of 6 moles H}_2\text{O}}{\text{mass of one mole FeCl}_3 \cdot 6\text{H}_2\text{O}} \times 100$$

$$\% \text{ water} = \frac{6 \text{ moles} \left(18.02 \frac{\text{g}}{\text{mol}} \right)}{55.85 \text{ g Fe} + (3 \times 35.45 \text{ g Cl}) + (6 \times 18.02 \text{ g H}_2\text{O})} \times 100$$

$$\% \text{ water} = \frac{108.12 \text{ g H}_2\text{O}}{270.32 \text{ g FeCl}_3 \cdot 6\text{H}_2\text{O}} \times 100 = 40.00\%$$

- Identify your unknown hydrated salt by comparing the average % water (by mass) for your unknown with the calculated theoretical % water for the salts listed on your data sheet.

Pre-Lab Questions

Prepare for lab by completing and understanding the answers to these questions. Refer to the Background or another resource, such as your textbook, if necessary.

1. Define the following terms: hydrate, desiccant, anhydrous, efflorescence, deliquescence
2. Name these compounds:
 - a. $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$
 - b. $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$
3. A sample of a hydrated salt weighing 1.048 g is heated until no more water is given off and a relatively constant weight is obtained. The mass of the anhydrous salt was 0.893 g. What is the mass of water lost by the sample? Calculate the mass % of water in the hydrated salt.
4. Calculate the mass % water in $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$. Report your answer to four significant figures.
5. Two of the possible unknowns in this lab are also commonly known as “gypsum” and “Epsom salts,” respectively. Do a little research (library, web) to identify these compounds and some of the applications in which they are used.

Lab 9: Water of Hydration Report Sheet

Name _____

Section _____

Date _____

Instructor _____

Water of Hydration

Data

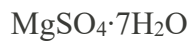
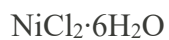
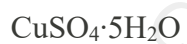
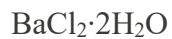
	Trial 1	Trial 2
Mass of crucible + lid (g)	_____	_____
Mass of crucible, lid, + hydrated salt (g)	_____	_____
Mass of crucible, lid, + anhydrous salt (g) (<i>weight 1</i>)	_____	_____
Mass of crucible, lid, + anhydrous salt (g) (<i>weight 2</i>)	_____	_____
Mass of crucible, lid, + anhydrous salt (g) (<i>weight 3</i>)	_____	_____

Calculations

	Trial 1	Trial 2
Mass of hydrated salt (g)	_____	_____
Mass of anhydrous salt (g)	_____	_____
Mass of H ₂ O lost (g)	_____	_____
% water in Unknown	_____	_____
Average % water	_____	
Identity	_____	

Observations:

Calculate the % H₂O for Possible Unknowns (show calculations)



Post-Lab Questions

1. Assuming that you have correctly identified your unknown, calculate the % error in your result and indicate if it is a positive or negative error. Discuss possible sources of error, paying attention to the direction of the error in each case.

2. Write the formula for your unknown and give its correct chemical name.

3. Did the appearance of your unknown change when it was dehydrated and rehydrated? Why do you think this is so?
4. Heating some hydrates too strongly may cause them to decompose. For example, some sulfates can emit SO_2 or CO_2 gas upon strong heating. If this were to occur in this experiment would the calculated mass % water be too low or too high? Explain.
5. A student takes 1.658 g of hydrated iron (III) nitrate and heats it in a crucible. The anhydrous material weighed 0.991 g. What is the formula and name for this hydrated salt? Show your work.

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