Unit 3 – Chapter 3 Experiment #13 Lab: Water of Hydration **Name ______________**

Period _____________

Most solid chemical compounds will contain some water if they have been exposed to the atmosphere for any length of time. In most cases, the water is present in very small amounts and is merely adsorbed on the surface of the crystals. Other solid compounds contain larger amounts of water that is chemically bound in the crystal. These compounds are usually ionic salts. The water that is present in these salts is called water of hydration and is usually bound to the cations in the salt.

The water molecules in a hydrate are removed relatively easily. In most cases, simply heating a hydrate to a temperature somewhat above the boiling point of water will drive off the water of hydration. Hydrated copper (II) chloride is typical in this regard; it is converted to anhydrous $CuCl_2$ if heated to about 110^0C: $CuCl_2 \cdot 2\ H_2O \rightarrow CuCl_{2(s)} + 2\ H_2O_{(g)}$ at $t \geq 110^0C$

In the dehydration reaction the crystal structure of the solid will change and the color of the salt may also change. On heating $CuCl_2 \cdot 2\ H_2O$ the green hydrated crystals are converted to a brownish-yellow powder. You may be familiar with hydrated $CoCl_2$, which is used in inexpensive hygrometers. $CoCl_2 \cdot 6\ H_2O$ is red, $CoCl_2 \cdot 2\ H_2O$ is violet, and $CoCl_2$ is blue.

Some hydrates lose water to the atmosphere upon standing. This process is called efflorescence. The amount of water lost depends on the amount of water in the air, as measured by its relative humidity. In moist, warm air, $CoCl_2$ is fully hydrated and is read; in dry, cold air $CoCl_2$ loses most of its water of hydration and is blue; at intermediate humidities $CoCl_2$ exists as a dihydrate and is violet.

Some anhydrous ionic compounds will tend to absorb water from the air or other sources so strongly that they can be used to dry liquids or gases. These substances are called **desiccants**, and are said to be hygroscopic. A few ionic compounds can take up so much water from the air that they dissolve in the water they absorb; sodium hydroxide, NaOH, will do this. This process is called **deliquescence**.

Some compounds evolve water on being heated but are not true hydrates. The water is produced by decomposition of the compound rather than by loss of water of hydration. Organic compounds, particularly carbohydrates, behave this way. Decompositions of this sort are not reversible; adding water to the product will not regenerate the original compound. True hydrates typically undergo reversible dehydration. Adding water to anhydrous $CuCl_2$ will cause formation of $CuCl_2 \cdot 2\ H_2O$ or, if enough water is added, you will get a solution containing hydrated Cu^{2+} ions. All ionic hydrates are soluble in water and are usually prepared by crystallization from water solution. The amount of bound water may depend upon the way the hydrate is prepared, but in general the number of moles of water per mole of ionic compound is either an integer or a multiple of ½.

In this experiment you will study some of the properties of hydrates. You will identify the hydrates in a group of compounds, observe the reversibility of the hydration reaction, and test some substances fro efflorescence or deliquescence. Finally you will be asked to determine the amount of water lost by a sample of unknown hydrate on heating. From this amount, if given the formula or the molar mass of the anhydrous sample, you be able to calculate the formula of the hydrate itself.

<u>Experimental Procedure</u> **Wear your safety classes while performing this experiment!**

A. Identification of Hydrates: Place about 0.5 g of each of the compounds listed below in small, dry test tubes, one compound to a tube. Carefully observe the behavior of each compound when you heat it gently with a burner flame. If droplets of water condense on the cool upper walls of the test tube, this is evidence that the compound may be a hydrate. Note the nature and color of the residue. Let the tube cool and try to dissolve the residue in a few cm^3 of water, warming very gently if necessary. A true hydrate will tend to dissolve in water, producing a solution with a color very similar to that of the original hydrate. If the compound is a carbohydrate, it will give off water on heating and will tend to char. The solution of the residue in water will often be caramel-colored.

Nickel chloride	Sucrose	Calcium carbonate
Potassium chloride	Sodium tetraborate (borax)	Barium chloride

B. Reversibility of Hydration: Gently heat a few crystals, ~0.3 g, of hydrated cobalt (II) chloride, $CoCl_2 \cdot 6\ H_2O$, in an evaporating dish until the color change appears to be complete. Dissolve the residue in the evaporating dish in a few cm^3 of water from your wash bottle. Heat the resulting solution to boiling (**CAUTION!**), and carefully boil it to dryness. Note any color changes. Put the evaporating dish on the lab bench and let it cool.

C. Deliquescence and Efflorescence: Place a few crystals of each of the compounds listed below on separate watch glasses and put them next to the dish of $CoCl_2$ prepared in Part B. Depending on their composition and the relative humidity (amount of moisture in the air), the samples may gradually lose water of hydration to, or pick up water from, the air. They may also remain unaffected. To establish whether the samples gain or lose mass, weigh each of them on a top-loading balance to 0.01 g. Record their masses. Weigh them again after about an hour to detect any change in mass. Observe the samples occasionally during the laboratory period, noting any changes in color, crystal structure, or degree of wetness that may occur.

$Na_2CO_3 \cdot 10\ H_2O$ (washing soda)	$KAl(SO_4)_2 \cdot 12\ H_2O$ (alum)
$CaCl_2$	$CuSO_4$

D. Percentage of Water in a Hydrate: Clean a porcelain crucible and its cover with 6 *M* HNO_3. Any stains that are not removed by this treatment will not interfere with this experiment. Rinse the crucible and cover with distilled water. Put the crucible with its cover slightly ajar on a clay triangle and heat with a burner flame, gently at first and then to redness for about 2 minutes. Allow the crucible and cover to cool, and then weigh them to 0.001 g on an analytical balance. Handle the crucible with clean crucible tongs.

Obtain a sample of unknown hydrate from the stockroom and place about a gram of sample in the crucible. Weigh the crucible, cover, and sample on the balance. Put the crucible on the clay triangle, with the cover in an off-center position to allow the escape of water vapor. Heat again, gently at first and then strongly, keeping the bottom of the crucible at red heat for about 10 minutes. Center the cover on the crucible and let it cool to room temperature. Weigh the cooled crucible along with its cover and contents.

Examine the solid residue. Add water until the crucible is 2/3 full and stir. Warm gently if the residue does not dissolve readily. Does the residue appear to be soluble in water? Solids can be thrown away.

DATA AND OBSERVATIONS: WATER OF HYDRATION

A. Identification of Hydrates

	H_2O appears	Color of residue	Water soluble	Hydrate
Nickel chloride				
Potassium chloride				
Sodium tetraborate				
Sucrose				
Calcium carbonate				
Barium chloride				

B. Reversibility of Hydration

Summarize your observations on $CoCl_2 \cdot 6\ H_2O$:

Is the dehydration and hydration of $CoCl_2$ reversible?

C. Deliquescence and Efflorescence

	Mass initial (sample + glass)	Mass final (sample + glass)	Observations	Conclusions
$Na_2CO_3 \cdot 10\ H_2O$				
$KAl(SO_4)_2 \cdot 12\ H_2O$				
$CaCl_2$				
$CuSO_4$				

D. Percent Water in a Hydrate

Mass of crucible and cover ____________________ g

Mass of crucible, cover, and solid hydrate ____________________ g

Mass of crucible, cover, and residue ____________________ g

Calculations and Results

Mass of solid hydrate ____________________ g

Mass of residue ____________________ g

Mass of H_2O lost ____________________ g

*Percentage of H_2O in the unknown hydrate ____________________ %

Formula mass of anhydrous salt (if furnished) ____________________

Number of grams H_2O per 100 g hydrate ____________________ g

Number of moles H_2O per 100 g hydrate ____________________ moles

Number of grams anhydrous salt per 100 g hydrate ____________________ g

Number of moles anhydrous salt per 100 g hydrate ____________________ moles

Formula of hydrate ($X \cdot n\ H_2O$, furnish *n* and X if given) ____________________

Unknown sample number ____________________