Name_____Lab Day_____Lab Time_____

Experiment 1 · Hydrates

Pre-lab questions

Answer these questions and hand them to the TF before beginning work.

(1) What is the purpose of this experiment?

_____

_____

(2) What is a hydrate?

_____

_____

(3) You will heat a crucible to "constant weight": how do you know that the crucible will have attained constant weight?

_____

_____

(4) You will drive off the water from a hydrate by heating it. What do you expect to see as the dehydration takes place? How will you know when the dehydration is complete?

_____

_____

(5) The slope m of the best straight line through the dehydration data has an associated uncertainty σ_m. How is the value of x in the hydrate $MB{\cdot}xH_2O$ related to m and to σ_m?

_____

_____

Hydrates

Even people who have never studied chemistry know that water is represented by the formula H_2O. But how are the formulas of chemical compounds established? One method involves determining the relative amount by weight of each element in the compound.

For example, let's say you raid an illegal drug lab and find a white powder that you suspect is cocaine. When you heat 1.00 g of the white powder, it decomposes into 0.40 g of carbon (C, $\mathcal{M}$ = 12.01 g/mol) and 0.60 g of water (H_2O, $\mathcal{M}$ = 18.02 g/mol). The number of moles of carbon in the sample is

$$\left(\frac{0.40 \text{ g C}}{}\right)\left(\frac{\text{mol C}}{12.01 \text{ g C}}\right) = 0.033 \text{ mol C}$$

and the number of moles of water in the sample is

$$\left(\frac{0.60 \text{ g } H_2O}{}\right)\left(\frac{\text{mol } H_2O}{18.02 \text{ g } H_2O}\right) = 0.033 \text{ mol } H_2O$$

There is one mole of carbon per mole of water in the sample. Thus, the formula of the white powder could be CH_2O, or $C_2H_4O_2$, or $C_3H_6O_2$, etc. Whatever the case, the white powder cannot possibly be cocaine, whose formula is $C_{17}H_{21}NO_4$; the white powder may be mannose ($C_6H_{12}O_6$), a sugar that drug dealers use to "cut" cocaine.

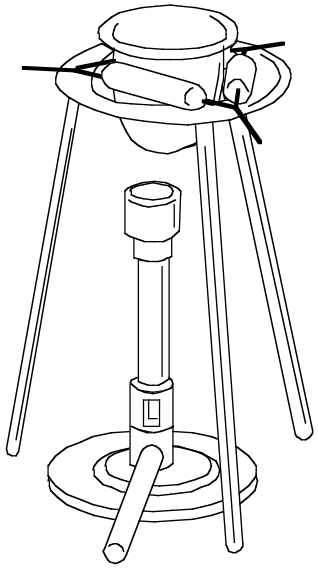
In this experiment we determine the formula of a hydrate. Hydrates are salts that incorporate water in their crystal structure. Simple hydrates have the generic formula $MB{\cdot}xH_2O(s)$, where M represents a positively charged ion (e.g., Cu^{2+}, Al^{3+} or Fe^{3+}), B represents a negatively charged ion (e.g., NO_3^-, SO_4^{2-}, or PO_4^{3-}) and *x* represents the number of moles of water that combine with each mole of MB in the hydrate. Many hydrates are familiar substances, ranging from everyday building materials such as gypsum ($CaSO_4{\cdot}2H_2O(s)$) to rare and expensive gemstones like turquoise ($CuAl_6(PO_4)_4(OH)_8{\cdot}4H_2O(s)$).

Procedure

Quantitative analysis

Wash a crucible with detergent and tap water and give it a final rinse with deionized water. Do not allow any water used to wash or rinse the crucible to run down the drain: collect it in a large beaker and, when the beaker is full, transfer its contents to one of the hazardous waste receptacles set up in the hoods.

Figure 1-1 Apparatus for heating a hydrate. The crucible, the Bunsen burner, the tripod and the wire triangle will be extremely hot after the heating period and will stay hot long after the flame is extinguished. Use tongs to handle the crucible; above all, do not touch any part of the apparatus with your bare hands.



Position the crucible inside a wire triangle supported by a tripod (see Figure 1-1). Heat the crucible over a Bunsen burner, let the crucible cool for 5 min and weigh it to two decimal places. Do not attempt to weigh the crucible if it is hot: the reading will be inaccurate and you risk damaging the balance. Do not touch the crucible with your bare hands after you have weighed it because the residue left by your fingerprints will change the crucible's weight. Instead, use tongs to handle the crucible. When moving about the lab, carry the crucible in a beaker to minimize the likelihood of accidentally dropping the crucible.

Heat the crucible a second time, let it cool for 5 min and reweigh it. If the two masses agree to within 0.05 g, the crucible has attained constant weight and you may continue with the experiment (assume that the lower weight is the crucible's true weight); otherwise, continue heating, cooling and weighing the crucible until constant weight as we have defined it is attained.

Once the crucible has attained constant weight, place a sample of hydrate whose mass ranges from 0.75 to 2.50 g in the crucible and record the mass of the crucible and sample to two decimal places. Be sure to record the identity of the hydrate. Heat the crucible and hydrate sample over a Bunsen burner until the dehydration appears to be complete. Discontinue heating, let the crucible cool for 5 min, then weigh the crucible and its contents to two decimal places. The dehydrated salt may be discarded in a trash can.

Repeat the entire procedure using the same hydrate, but this time take a sample of significantly different mass. Yes, you must clean the crucible again with detergent and tap water and heat the crucible to constant weight as before.

Find at least four other groups who dehydrated the same substance you did and exchange data with them. Get from them (1) the two masses of hydrate they heated and (2) the two masses of dehydrated salt remaining after heating. You should have a total of ten data items including two of your own. Make sure that the sample sizes of hydrate span the entire range of 0.75 to 2.50 g.

Don't point the test tube you're heating at yourself or at others: the water may escape from the hydrate violently, ejecting hot material.

Do not add water to the test tube while the test tube is still hot: the mixture may boil over and you may burn your hands.

Qualitative observations

Place copper(II) sulfate hydrate ($CuSO_4 \cdot xH_2O(s)$), nickel(II) sulfate hydrate ($NiSO_4 \cdot xH_2O(s)$) and cobalt(II) chloride hydrate ($CoCl_2 \cdot xH_2O(s)$) in three separate 150-mm (big) Pyrex test tubes to a depth of no more than 1 cm. Record in your notebook the appearance of each hydrate. Heat each test tube over a Bunsen burner for a few minutes, holding the test tube at an angle and moving it in and out of the flame. Record what happens to each hydrate as you heat it. Discontinue heating, let the test tube cool, then add a few drops of deionized water to each test tube and record your observations.

Clean-up

The 150-mm test tubes are not disposable. Try to rinse out the residue remaining in each test tube by adding a few milliliters of water. Be sure to catch the rinse water in a hazardous waste receptacle: do not dump it down the drain. Place the rinsed test tubes in the test tube tray in the hood. The baked-on gunk may not wash out: that's OK, just try to clean up the test tubes as best you can.

Data analysis

The goal here is to extract the value of *x*, the number of moles of water per mole of hydrate in $MB \cdot xH_2O(s)$. Before *x* can be

Table 1-1 Data collected for the dehydration of strontium chloride hydrate ($SrCl_2 \cdot xH_2O(s)$)

Before heating	**Run 1**	**Run 2**
Mass of crucible	27.29 g	27.51 g
Mass of crucible + hydrate	28.56 g	30.00 g
Mass of hydrate[a]	1.27 g	2.49 g
After heating	**Run 1**	**Run 2**
Mass of crucible + dehydrated salt	28.05 g	28.99 g
Mass of dehydrated salt[a]	0.76 g	1.48 g
Mass of water lost	0.51 g	1.01 g

a This data item for your hydrate should be exchanged with at least four other students that dehydrated the same substance that you used.

evaluated, we must calculate (1) the number of moles of dehydrated salt remaining after heating and (2) the number of moles of water driven off during heating.

Table 1-1 shows sample raw data relating to the dehydration of strontium(II) chloride hydrate ($SrCl_2 \cdot xH_2O(s)$). By consulting the periodic table, we compute the molar mass of $SrCl_2$ ($\mathcal{M}$ = 158.52 g/mol) and that of water ($\mathcal{M}$ = 18.02 g/mol). If we assume that all the water is driven off during heating, that is, if we assume that the reaction

$$SrCl_2 \cdot xH_2O(s) \rightarrow SrCl_2(s) + x\ H_2O(g)$$

goes to completion, the number of moles of dehydrated salt remaining after heating in Run 1 is found as follows:

$$\left(\frac{0.76\text{ g salt}}{}\right)\left(\frac{\text{mol salt}}{158.52\text{ g salt}}\right) = 0.0048\text{ mol salt}$$

Likewise, the number of moles of water that are driven off during the heating process in Run 1 is given by

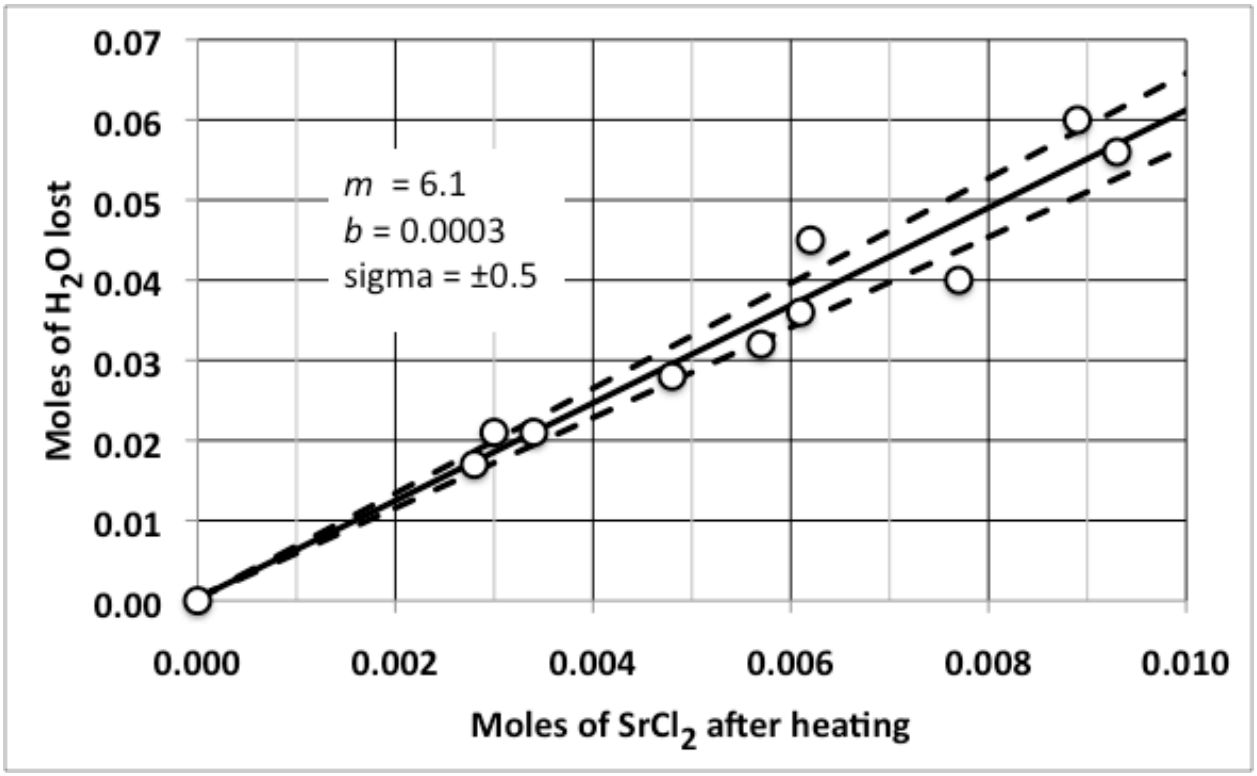
$$\left(\frac{0.51\text{ g } H_2O}{}\right)\left(\frac{\text{mol } H_2O}{18.02\text{ g } H_2O}\right) = 0.028\text{ mol } H_2O$$

Carry out calculations of this sort on your own data and on the data gathered from other students, then plot all the data, plotting the moles of dehydrated salt remaining after heating on the horizontal axis and the moles of water lost on the vertical axis; include the origin {0,0} as a data point.

Determine the slope m and the y-intercept b of the best straight line through the data by the method of linear least-squares given in Appendix A "Statistical Treatment of Data" of this lab manual; include the origin {0,0} as a data pair in the calculations. Draw the least-squares line through the data. Also calculate the standard error of estimate σ_m in the slope. Appendix A gives directions for calculating m, b and σ_m using a calculator and using the Microsoft® Excel® spreadsheet application. You may use either method, but Excel® makes the task easier and faster.

The value of x in the hydrate $MB{\cdot}xH_2O(s)$ corresponds to whatever integer(s) lie within the interval $m - \sigma_m$ and $m + \sigma_m$. On your plot, be sure to draw the two lines $y = (m - \sigma_m)x + b$ and $y = (m + \sigma_m)x + b$ that define the uncertainty inherent in the data. Your plot should resemble Figure 1-2, especially in terms of clarity.

Figure 1-2 Determination of the number of moles of water per mole of $SrCl_2$ in $SrCl2{\cdot}xH_2O(s)$. The slope m of the line determined by the method of least squares is 6.1 mol H_2O/mol $SrCl_2$ (solid line); the standard error of estimate σ_m in the slope is ±0.5 mol H_2O/mol $SrCl_2$ (dashed lines defining the uncertainty in the slope of the least-squares line). Thus, the data indicate that x in $SrCl_2{\cdot}xH_2O(s)$ lies between 6.1 – 0.5 = 5.6 and 6.1 + 0.5 = 6.6, that is, the data suggest the formula $SrCl_2{\cdot}6H_2O(s)$.



Name_____Lab Day_____Lab Time_____

Experiment 1 · Hydrates

Lab report form **Page 1**

(I) Report the quantitative data relating to the dehydration. Enter the data you collected in the first two lines of the table below and the data collected from other groups in the remaining lines.

Hydrate = _____

Molar mass $\mathcal{M}$ of the salt in the hydrate = _____ g/mol

Run	**Mass of hydrate [g]**	**Mass of dehydrated salt [g]**	**Mass of H_2O lost [g]**	**Moles of dehydrated salt**	**Moles of H_2O lost**
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

(II) Calculate the linear least-squares slope m, y-intercept b and standard error of estimate σ_m using the method of linear least-squares given in Appendix A "Statistical Treatment of Data" of this lab manual. Include the origin {0,0} as a data point in all calculations.

Linear least-squares slope m = _____ mol H_2O/mol salt

Linear least-squares y-intercept b = _____ mol H_2O

Standard error of estimate σ_m = _____ mol H_2O/mol salt

Name_____Lab Day_____Lab Time_____

Experiment 1 · Hydrates

Lab report form **Page 2**

(III) On a separate sheet, prepare a plot of the data similar to Figure 1-2: label the axes and include appropriate units and divisions of those axes. Do not submit a small plot: use the whole sheet of paper. Scale the horizontal and vertical axes so that the data points occupy most of the area of the plot. Draw the linear least-squares line through the data, and draw the two lines $y = (m \pm \sigma_m)x + b$ that describe the uncertainty in the linear least-squares slope. Don't forget to plot the origin {0,0} and don't forget to use the origin as a data point in all calculations. Write your values of m, b and σ_m on the plot. Use Figure 1-2 as your guide: the plot should be neat and easy to read; try to make it look beautiful.

(IV) Report the appearance of the hydrates that you heated in the small test tubes before heating, after heating and upon the addition of water after heating.

Hydrate	**Before heating**	**After heating**	**Upon adding H_2O**
$CuSO_4 \cdot xH_2O$			
$NiSO_4 \cdot xH_2O$			
$CoCl_2 \cdot xH_2O$			

Name_____Lab Day_____Lab Time_____

Experiment 1 · Hydrates

Lab report form **Page 3**

Post-lab questions

(1) Write the balanced chemical equation of the reaction that takes place when water is driven off from the hydrate for which you determined the value of *x*.

(2) Is the data sufficiently reliable to have firmly established the value of *x*? Explain your answer.

(3) Suppose that a student reports a value of *x* that is lower than what the actual value of *x* is supposed to be. What operation did the student most likely execute incorrectly in the lab to cause this error? Assume that no computational blunders were made.

Name_____Lab Day_____Lab Time_____

Experiment 1 · Hydrates

(4) When water is added to the salts that were heated in the small test tubes, the original color of the hydrate returns in some cases, but not in others. Explain why the original color does not return.

(5) Kalinite ($KAl(SO_4)_2 \cdot 12H_2O(s)$, $\mathcal{M}$ = 474.38 g/mol) is a hydrate found in volcanic ash. A 14.52-g sample of volcanic ash containing kalinite is heated until all the water in the kalinite is driven off. At the end of the heating period, the sample weighs 14.18 g. Assuming that all the water driven off in the sample comes from kalinite, calculate the mass of kalinite in the original sample; show all calculations.