

Lab Session 5, Experiment 4: Law of Definite Proportions

The law of definite proportions states that when two or more elements combine to form a given compound, they do so in fixed proportions by mass. This is the same as saying the composition of a compound is fixed. For example, sodium chloride contains 39.3% by mass sodium and 60.7% by mass chlorine.

In this experiment a sample of KClO_3 will be decomposed thermally, and the oxygen produced will be expressed as a percentage of the original mass of KClO_3 . Experimental results will then be compared to the theoretical percentage of oxygen in KClO_3 .

4A Experiment: Decomposition of a Salt

1. Add approximately 0.1 g of MnO_2 to a clean, dry crucible and weigh the crucible including the catalyst. Enter the mass on line (b) below.
2. Add KClO_3 to the crucible until it is about one-third full. Mix the KClO_3 and MnO_2 thoroughly. Weigh again and enter the mass on line (a) below.
3. Place the crucible on a wire triangle supported on a ring clamp. Adjust the ring clamp to a height that allows the crucible to be heated with the flame of your burner.
4. Heat the crucible slowly at first. Then regulate heating to prevent boil-over from the crucible. The objective is to completely decompose the chlorate to the chloride and oxygen; therefore, heat for about ten minutes after apparent total decomposition. Heating should be so vigorous as to cause the bottom of the crucible to glow red. Heat until all purple color is gone and the contents are a uniform gray color.
5. Allow the crucible to cool. Weigh the crucible and enter the mass on line (d) below.
6. Compute the following: mass of oxygen, mass of KCl , experimental percentage of oxygen, theoretical percentage of oxygen, and percent error in the experimental percentage of oxygen, calculated as $[(\text{actual yield} - \text{theoretical yield}) / \text{theoretical yield}] \times 100$.

(a) Mass of crucible with MnO_2 and KClO_3	g
(b) Mass of crucible with MnO_2	g
(c) Mass of KClO_3 [(a)–(b)]	g
(d) Mass of crucible and contents after heating	g
(e) Mass of oxygen [(a)–(d)]	g
(f) Mass of KCl [(c)–(e)]	g
(g) Experimental percentage of oxygen	%
(h) Theoretical percentage of oxygen	%
(i) Percent error in experimental percentage of oxygen	%

7. Now assume that the compound you decomposed has the formula KClO_x . Note that "x" is the ratio of moles of O to moles of KCl . Complete the following table.

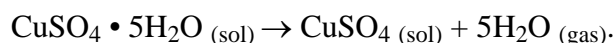
(j) Experimental moles of O	mol
(k) Experimental moles of KCl	mol
(l) Calculated value of above ratio	
(m) Formula of compound decomposed	

Hydrates

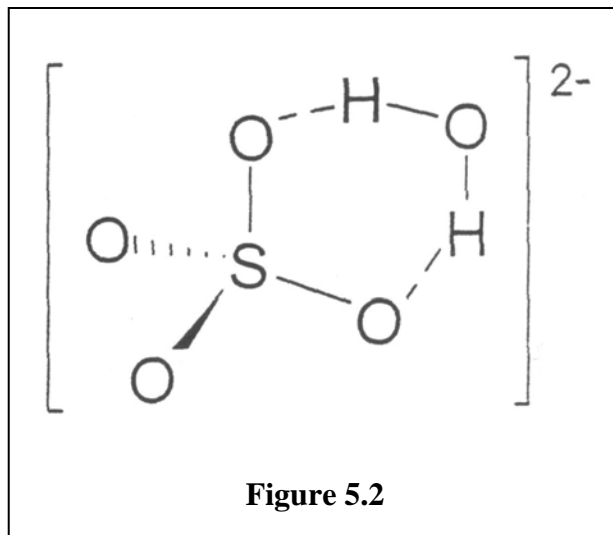
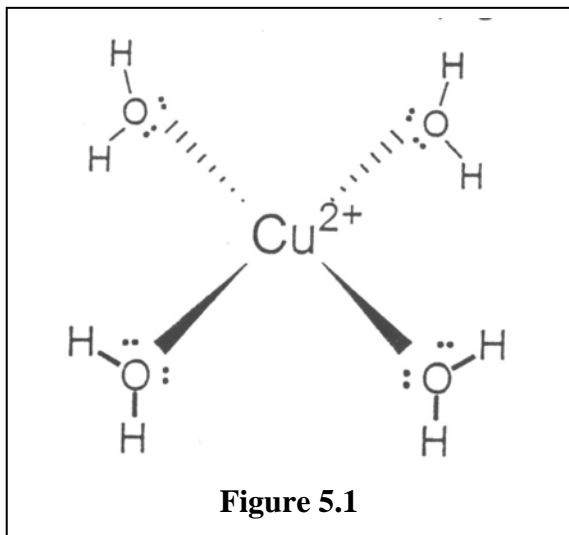
Hydrates are substances formed when water combines chemically in definite proportions with a salt. The ratio of water molecules to the ions of the salt is a constant. Hydrates are not mixtures. The anhydrous (without water) form of the salt is produced when all the water of hydration is lost. Some examples of hydrates are listed below:

<u>Formula</u>	<u>Names</u>
$(\text{CaSO}_4)_2 \cdot \text{H}_2\text{O}$	calcium sulfate hemihydrate "plaster of paris"
$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	calcium sulfate dihydrate "gypsum"
$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	copper (II) sulfate pentahydrate "blue vitriol"
$\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	magnesium sulfate heptahydrate "epsom salt"
$\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$	sodium carbonate decahydrate "washing soda"

The • in the formula indicates a kind of chemical bond that usually can be easily broken. For example, copper (II) sulfate pentahydrate can be converted to anhydrous copper (II) sulfate by heating:



In $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ the bonding involves four water molecules coordinatively bound to the Cu^{2+} ion in a square planar structure (Figure 5.1) and one molecule of water bound to the sulfate ion by hydrogen bonds (Figure 5.2).



Loss of hydration water to the atmosphere is called **efflorescence**. The property of some salts to collect moisture from the air and dissolve in it is called **deliquescence**. A compound is **hygroscopic** if absorption of water from the atmosphere occurs without dissolution of the compound.

4B Experiment: Composition of a Hydrate

In the following experiment, record all masses in the data table.

1. Weigh your evaporating dish.
2. Introduce about 5 g of pulverized hydrated CuSO_4 . Note the appearance and color of the solid; weigh the dish and contents.
3. Place the evaporating dish on the wire gauze and heat slowly until the color disappears. Prolonged heating may result in the decomposition of the anhydride. What color change occurred? _____
4. When cool, weigh the dish and anhydride.
5. From the data, calculate the following:
 the percentage of water in the hydrate $[(\text{mass of H}_2\text{O}) \div (\text{mass of hydrate})] \times 100$,
 the number of moles of water $[(\text{mass of H}_2\text{O}) \div \text{molar mass of H}_2\text{O}]$,
 the number of moles of anhydride $[\text{mass of anhydride} \div \text{molar mass of anhydride}]$, and
 the formula of the hydrate $\text{CuSO}_4 \cdot x \text{H}_2\text{O}$ where $x = [(\text{moles of water}) \div (\text{moles of anhydride})]$.
6. Add water a few drops at a time to convert the anhydride back to the hydrate. Do you notice a change in temperature? (Check with a thermometer.) _____
7. What evidence of a chemical change did you observe? _____

	Formula of Copper Sulfate Hydrate	
	Experiment	Duplicate
(a) Mass of crucible or dish and hydrate	g	g
(b) Mass of empty crucible or dish	g	g
(c) Mass of hydrate [(a) – (b)]	g	g
(d) Mass of crucible or dish and anhydride	g	g
(e) Mass of anhydride [(d) – (b)]	g	g
(f) Mass of water in hydrate [(c) – (e)]	g	g
(g) Percentage of water in hydrate [(f) ÷ (c)] × 100	%	%
(h) Moles of water [(f) ÷ molar mass of H ₂ O]	mol	mol
(i) Moles of anhydride [(e) ÷ molar mass of anhydride]	mol	mol
(j) Moles of water per mole of anhydride [(h) ÷ (i)]		
(k) Theoretical moles of water per mole of anhydride		
(l) % error $[((j) - (k)) \div (k)] \times 100$		
(m) Formula of hydrate		
(n) Equation for decomposition of your hydrate		

4C Experiment: Properties of hydrates

1. Place crystals of calcium chloride, washing soda, and lithium chloride on a watch glass and expose them to the air of the laboratory.
 2. Note the appearance of each sample towards the end of the lab. Which crystals deliquesce?
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Which crystals effloresce?

Explain why one sample gained water and why one lost water.

4D Experiment

1. Introduce one spatula of each of the following into separate test tubes: sodium chloride, potassium dichromate, aluminum sulfate, cobalt chloride, and nickel sulfate. Record color before heating in the table below.
 2. Heat each sample in turn, holding the tube at an angle of about 30° so that any water will condense in the cool part of the tube. Record color after heating in the table below.
 3. When you heated salts in this experiment, which ones lost water?
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4. Can the original color be restored by addition of a few drops of water to the cooled tube?
-

List any hydrates that changed color upon heating, but for which the color came back when water was added.

	Color Before Heating	Color After Heating
(a) Sodium Chloride		
(b) Potassium Dichromate		
(c) Aluminum Sulfate		
(d) Cobalt Chloride		
(e) Nickel Sulfate		

Report Form 4: Law of Definite Proportions

Name _____
Partner _____ Section # _____

4A Experiment: Decomposition of a Salt

(a) Mass of crucible with MnO ₂ and KClO ₃	g
(b) Mass of crucible with MnO ₂	g
(c) Mass of KClO ₃ [(a)–(b)]	g
(d) Mass crucible and contents after heating	g
(e) Mass of oxygen [(a)–(d)]	g
(f) Mass of KCl [(c)–(e)]	g
(g) Experimental percentage of oxygen	%
(h) Theoretical percentage of oxygen	%
(i) Percent error in experimental percentage of oxygen	%
(j) Experimental moles of O	mol
(k) Experimental moles of KCl	mol
(l) Calculated value of above ratio	
(m) Formula of compound decomposed	

4B Experiment: Composition of a Hydrate

What color change occurred? _____

Do you notice a change in temperature? (Check with a thermometer.) _____

What evidence of a chemical change did you observe? _____

	Experiment	Duplicate
(a) Mass of crucible or dish and hydrate	g	g
(b) Mass of empty crucible or dish	g	g
(c) Mass of hydrate [(a) – (b)]	g	g
(d) Mass of crucible or dish and anhydride	g	g
(e) Mass of anhydride [(d) – (b)]	g	g
(f) Mass of water in hydrate [(c) – (e)]	g	g
(g) Percentage of water in hydrate [(f) ÷ (c)] × 100	%	%
(h) Moles of water [(f) ÷ molar mass of water]	mol	mol
(i) Moles of anhydride [(e) ÷ molar mass of anhydride]	mol	mol
(j) Moles of water per mole of anhydride [(h) ÷ (i)]		
(k) Theoretical moles of water per mole of anhydride		
(l) % error [((j) – (k) ÷ (k)) × 100]		
(m) Formula of hydrate		
(n) Equation for decomposition of your hydrate		

4C Experiment: Properties of hydrates

Which crystals deliquesce? _____

Which crystals effloresce? _____

Explain why one sample gained water and why one lost water. _____

4D Experiment

When you heated salts in this experiment, which ones lost water?

Can the original color be restored by addition of a few drops of water to the cooled tube?

List any hydrates that changed color upon heating, but for which the color came back when water was added. _____

	Color Before Heating	Color After Heating
(a) Sodium Chloride		
(b) Potassium Dichromate		
(c) Aluminum Sulfate		
(d) Cobalt Chloride		
(e) Nickel Sulfate		