Experiment 1
Stoichiometry : Solids
Determining the Formula of a Compound

Introduction:

The Mole

As with any area of technical expertise, chemists have a unit of quantity, known as the mole. It is no different from, say, a unit of length; e.g. a meter. Having distance in meters allows one to convert it to any other unit of length, by simply looking up the relevant conversion factor. So too with the mole, once it is known for any substance in a given chemical reaction, then the quantity of all other substances can be derived by using the appropriate conversion factor. In our case, these conversions are dependent upon the reaction in question and can only be arrived at using a balanced chemical reaction. Coefficients before each substance will be used to make our conversion factors.

Why use the mole(?), since the fundamental unit of an element or a compound is an atom or molecule, respectively. Why not use the mass of the individual atom or the sum of the atoms that make up the molecule? Size is the issue here, the mass of an atom is so small (~10^{-23} g) that, while it is possible to determine, it is impractical to do so in most laboratory experiments. Somewhat analogous to the fact that when you refer to the distance from Amherst to Northampton you speak in terms of ~10miles rather than ~1,600,000 cms! A more practical mass range from the laboratory perspective is in the gram range, 1-300g. In order to define the mole in terms of the fundamental unit (atom/molecule), a standard had to be chosen, and this turned out to be the number of atoms in twelve grams of pure carbon twelve (an isotope of carbon with mass number 12). The number of atoms turns out to be 6.023×10^{23} , which has come to be known as Avogadro's Number. Apart from its large size there is nothing special about this number, and if a different standard were chosen it would have a different value. A mole can thus be redefined as the mass of any substance that contains 6.023×10^{23} units of that substance. Its use is pretty much confined to atoms and molecules and the unit is hardly likely to replace the dozen when it comes to large objects such as eggs! The mass of any element that corresponds to 6.023×10^{23} atoms of that element is equal to its molar mass, which for an element can be found in the periodic table. The masses listed on the table are in fact average values, derived from the various isotopes of the elements and their natural abundances. For molecules, the molar mass is the sum of the individual atom masses that make up the molecule.

In summary: 1 mole is equal to 6.023×10^{23} atoms (or molecules) which in turn is equal to the atomic mass of the element or the molar mass of the molecule in grams .

In the laboratory, from a stoichiometric point of view, you can classify the substances that you will be dealing with as either, solids, solutions, or gases. For this experiment we will be dealing exclusively with solids where:

moles = mass (in grams) /molar mass

Determining the Formula of a Compound

One of the most exciting aspects of chemistry is the discovery of some new substance when one element reacts with another. In order to know whether one has made a new substance, the formula of the compound formed must be determined quantitatively. This involves determining the mole ratio and not the gram ratio. The gram ratio gives no meaningful information since the individual masses are not related to equivalent number of atoms.

While the experiment that you will be performing in this laboratory is not a new discovery, we are going to treat it as such! You will burn a measured quantity of magnesium in the presence of air (your source of oxygen) and then use mass measurements to determine a possible formula for the resultant product:

$$x\ \text{Mg(atoms)} + y\ \text{O (atoms)} \implies Mg_xO_y\ \text{(molecule)}$$

> **Note:**
> *When no integer appears before a atom or molecule in the balanced chemical equation, it is inferred that only one molecule is produced, thus the product of the above reaction is written as Mg_xO_y and not 1 Mg_xO_y.*

The amount of magnesium is readily determined by weighing the initial amount of it. Assuming that one gets complete conversion to the product Mg_xO_y, then by weighing the amount of product produced, the amount of oxygen that reacted can be determined (mass of the product - mass of magnesium used). Knowing the mass of each, we can convert these masses to moles, and then determine the mole ratio of magnesium to oxygen.

Remember that an atom is the smallest particle of an element that still retains the chemical properties of that element. Thus the mole ratio should turn out to be a whole number or by simple multiplication can be transformed into a whole number. However the resultant formula is not necessarily the actual formula since all that we have determined is the ratio of one atom to the other, e.g.

Mg/O = 1:1, could be MgO, Mg_2O_2, Mg_3O_3……etc, i.e. any combination whose atom ratio is 1:1

What if the mole ratio is not an integer?..i.e. Mg/O = 1.66:1. The temptation is to round the 1.66 up 2.00 and thus obtain a ratio of 2:1. Do not do this! One method is to express the number as a fraction, i.e. 1.66 = 5/3 and thus by multiplying the ratio by 3 one would get a ratio 5:3. Another way is to multiply the ratio in increments of one until you obtain a whole number ratio:

Mg/O = 1.66 : 1 = 2(1:66 : 1) = 3.32 : 2 = 3(1.66 : 1) = 4.98 : 3

It is now safe to round 4.98 to 5 and obtain 5:3. This latter method may look cumbersome, but it works all the time and it does not depend on you recognizing fractions!

Mg/O = 5:3, could be Mg_5O_3, $Mg_{10}O_6$ ….etc, again any combination whose atom ratio is 5:3.

Note that your experimental ratio is unlikely to be exactly 1:1 or 1.66/1 or any other whole number or simple fraction. [Why?]

The formula based on the simplest mole ratio is referred to as the empirical formula. In order to get the actual formula we would need one more piece of data, the actual molar mass of the compound.

Recording Data and Significant Figures

Significant Figures:

Since this is your first experiment, and many of the experiments that you will be performing are quantitative in nature (meaning the result is a determined number), let's delve briefly into significant figures. In most experiments many different kinds of measurements are made using a variety of different tools, some of which measure to a higher degree of precision than others. It then makes sense that a calculated result based on these measurements can be no more precise than the least precise measurement that went into that calculation. This is the essence of significant figures.

1. In adding or subtracting measurements, the number of decimal places in the answer should equal the measurement with the fewest decimal places.
2. In determining the number of significant figures in a measurement, read from left to right and count all digits, starting with the first digit that is not zero: e.g.

    1. 0.049g: 2 Significant figures
    2. 2.0g: 2 Significant figures

    Constants are considered to have infinite number of significant figures. Put in another way, these quantities should not be used in determining the measurement with the least number of significant figures.

3. In multiplication and division, the number of significant figures in the calculated quantity should be the same as the quantity used with the fewest number of significant figures.

    Note:
    When using a calculator, do the calculation using all the digits allowed by the calculator and round off the final answer.

Rounding: round up if the digit after the last significant figure equals or is greater than 5.

With regards to the laboratory only:

- you may treat as a constant any reagent concentrations given in an experiment.
- if in doubt use one more rather than one less significant figure.
- we are primarily concerned with significant figures only in the final answers. With all other data we are concerned with recording the data to the accuracy of the instrument used.

Recording Data:

This is one area where we place a lot of emphasis. Always record the data so that it reflects the accuracy of the instrument used. With digital instruments that simply means recording all the digits displayed, even zeros!

With other equipment you record to 1/10th or 1/5th of the smallest division. In the case of a buret, the smallest division is 0.1 therefore you should record to two decimal places in increments of 0.01 or 0.02 depending on you ability to guesstimate! With a 400mL beaker, the smallest division is 25mL, therefore you should record this in increments of either 2.5mL or 5mL.

Experimental Procedure:

Using an Electronic Balance

To determine mass you have three models of electronic balances available to you. These balances measure to 0.001g (1mg) and when you use them you should always record all three decimal places. The S.I. unit for mass is the kilogram (1 kg = 1000g). However it is not common to express mass in the laboratory in kilograms.

Be aware that these balances are somewhat delicate and very sensitive. Do not remove the plastic guard around each. While it may not always be practical to do so, it is good practice to stick with the same balance during any particular experiment. Pictured are the three models of analytical balances that we use in the General Chemistry. During the first lab, your TA will demonstrate the various idiosyncrasies of each! When doing any weighing, please observe the following:

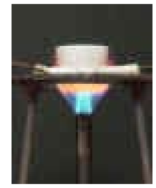
1. Do not remove the plastic guard around each balance.
2. Do not move the balance, this throws off the calibration and necessitates recalibration of the balance.
3. Weigh chemicals in containers that are dry on the outside. Never weigh directly on the pan.
4. Weigh everything at room temperature.
5. Zero the balance and keep the draft doors closed during weighing. Avoid leaning on the benches as this in fact interferes with the weighing!

PLEASE LEAVE THE BALANCE CLEANER THAN YOU FOUND IT. Brushes have been attached to each balance to facilitate this.

Determining the Formula of a Compound

1. Wash a crucible with soap, rinse with tap water and finally distilled water. You will not be able to get the inside immaculately clean but anything that remains after washing thoroughly and heating will not affect the reaction.

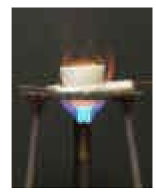
2. 
Adjust the flame on your Bunsen Burner such that it resembles the picture on the left. This blue flame is needed to generate enough heat so as to ignite the magnesium. A yellow flame would not only have difficulty in starting the reaction but would also deposit carbon on the crucible thus destroying any hope of obtaining meaningful results.

3. Support the crucible on a tripod using a clay triangle, as depicted.


   Adjust the height of the Bunsen Burner such that the tip of the inner blue flame touches the crucible. Heat the crucible for two to three minutes. If the burner is in the correct position the base of the crucible should glow orange.

4. When cool, handling the crucible with a paper towel, place it on top of a 50mL beaker and weigh it on the analytical balance (Do not place anything hot directly on the balances). Only weigh the crucible and beaker.

5. 
Cut a piece of magnesium equivalent to ~0.2-0.25g. Magnesium develops a coating of magnesium oxide over time. To remove this use the sand paper provided. Please do not do this on the bench top as sand paper leaves unsightly marks!!. Once you have sand papered the magnesium, roll it such that when it is placed in the crucible it sits on the bottom. This make ignition much easier Reweigh and record the mass of the crucible and the magnesium..

6. Heat crucible and its contents with a vigorous blue flame as depicted on the right. Once the bottom of the crucible starts to glow orange/red the magnesium should ignite very shortly after this. This should take no more than five minutes to accomplish. If the magnesium does not ignite in this period of time you will have to start over as in all likelihood the magnesium metal has being coated with a oxide layer and will not ignite.


7. 
Once the magnesium has started to glow, you should remove the bunsen burner until the glowing has stopped. One it has stopped place the burner back under the crucible and continue to heat for a further 5 minutes. Remove the heat and allow the crucible to cool to room temperature.

   As we are using air as our source of oxygen (remember air is predominantly nitrogen) we may form some magnesium nitride and we need to convert any of this formed to the oxide. Hence step 8.

8. Carefully add 10 drops of distilled water to the crucible, once it has cooled, using a dropper. Make sure that all the white powder stays inside the crucible.

9. Heat using a blue flame for <u>five</u> minutes. Allow to cool. Weigh and record.
10. Continue this heating, cooling, re-weighing, until you get two readings that do not differ by more than 0.001g.
11. Repeat the whole procedure with a second sample.
12. Determine the moles of magnesium and oxygen atoms that reacted and then the mole ratio.

Chem 111 **Experiment 1** **Fall 2008**

Stoichiometry…Solids: Determining the Formula of a Compound

Name: ______________________ **Lab TA:** ______________________

Lab Day	**Mon**	**Tue(am)**	**Tue(pm)**	**Wed**	**Thu(am)**	**Thur(pm)**	**Fri**
	☐	☐	☐	☐	☐	☐	☐

Grade

	>95	**>90**	**>85**	**>80**	**>70**	**<70**
Report:						

Prelaboratory Quiz Score: ☐

Data Collection and Calculations:

			Trial 1	**Trial 2**
1.	a)	Mass of:		
		Crucible and beaker		
		Crucible, beaker and magnesium		
		Magnesium		
	b)	Moles of magnesium		
2.	a)	Mass of crucible, beaker and magnesium oxide:		
		After 1^{st} Heating		
		After 2^{nd} Heating		
		After 3^{rd} Heating (if required)		
	b)	Mass of magnesium oxide		
	c)	Mass of oxygen reacted		
	d)	Moles of oxygen reacted		
3.	a)	Ratio of moles of magnesium to moles of oxygen		
	b)	Average ratio of moles of magnesium to moles of oxygen		
	c)	Empirical Formula		
4.		Comment on the empirical formula you determined. Does it look reasonable? Why?		

Show Calculations for:

1b. Moles of magnesium.	**2d.** Moles of oxygen atoms reacted.

Post Laboratory Question:

Your TA will not help you with this final question.

A compound containing only xenon and fluorine can be obtained by shining light on a flask that contains 0.526g of xenon and excess fluorine gas. When all the xenon was consumed, 0.678g of the new compound was obtained. Determine the empirical formula of the compound?

Molar Masses:

Mg: 24.31g/mol O: 16.00g/mol Xe: 131.29g/mol F: 18.99g/mol