Lab 3: Quantitative Chemical Analysis:  
Determination of Relative Atomic Mass

Short, written answers are required to the questions below and are designed to enhance the students' awareness of some of the procedural details of the laboratory. They will be checked as the students enter the laboratory and count for 1 of 5 points for the laboratory exercise.

1. Give a brief explanation for how the mass of oxygen that has reacted with the magnesium is determined.

2. The magnesium ribbon used in the experiment is coiled sufficiently so as to fit in the bottom of the crucible. Why is it important that it is not coiled too tightly?

3. What purpose is served by the initial heating of the crucible and why is it important?

4. Describe the procedure for cooling the crucible.

5. The metal used in this experiment is the element magnesium. Research the element magnesium (metallic magnesium, not a compound of magnesium) and report a common use of it.

6. Why is it important to turn on the gas only when you are immediately ready to light it?
**Introduction**

One of the earliest triumphs for the atomic theory of matter was the simple manner in which it explained the curious observation of constant composition in compounds. Constant composition refers to the fact that the mass ratio of the elements contained in a compound is constant for that compound. For example, pure water is always found to be 88.9% oxygen and 11.1% hydrogen, whereas hydrogen peroxide, another compound formed from hydrogen and oxygen with distinctly different properties, is always 95% oxygen and 5% hydrogen; any other ratio is not possible in a pure compound containing only hydrogen and oxygen. Atomic theory rationalizes constant composition based primarily on three assumptions:

1. All atoms of a given element are identical in mass.
2. All atoms of different elements are different in mass.
3. Compounds contain the atoms of two or more elements in fixed, small whole number ratios.

Determination of the constant composition of a compound and subsequent calculation of the relative atomic masses is readily accomplished for metal oxides, a class of binary compounds (one containing two elements) formed from the reaction of a metal and oxygen. Many metals form the oxide merely by heating the metal in air. The constant composition is determined using the analysis outlined below.

\[
\text{Metal} + \text{Oxygen} \rightarrow \text{Metal Oxide}
\]

\[
\text{mass 1} + (\text{mass 2} - \text{mass 1}) \rightarrow \text{mass 2}
\]

Magnesium may be converted to magnesium oxide at temperatures safe in an undergraduate lab. The conversion is complicated somewhat by the simultaneous reaction of magnesium with atmospheric nitrogen. Fortunately, this product (magnesium nitride) is readily converted to the desired magnesium oxide by the addition of water and further heating. The complete series of chemical reactions is shown below:

1. \( \text{magnesium} + \text{oxygen} \rightarrow \text{magnesium oxide} \)
2. \( \text{magnesium} + \text{nitrogen} \rightarrow \text{magnesium nitride} \)
3. \( \text{magnesium nitride} + \text{water} \rightarrow \text{magnesium hydroxide} + \text{ammonia} \)
4. \( \text{magnesium hydroxide} \rightarrow \text{magnesium oxide} + \text{water} \)

Reactions 1 and 2 occur simultaneously when the magnesium is heated initially. The reaction vessel is then cooled and a small amount of water is added. Subsequent additional heating serves to drive reactions 3 and 4 as well as evaporate the ammonia and remaining water. Importantly, the magnesium is completely converted to pure magnesium oxide.

Calculation of the relative atomic mass, as described in the lecture, requires the combining ratio of magnesium and oxygen atoms. In the Conclusion and Discussion
section of the lab, we will assume several ratios and make a conclusion about which assumption is correct by referring to the periodic table.

**Procedural Overview**

The conversion of magnesium to magnesium oxide is accomplished by supporting a porcelain crucible containing the magnesium on a ring stand over a Bunsen burner, as shown in Figure 3.1. It is vital for the success of the experiment that the flame be adjusted properly; the use of the Bunsen burner will be discussed by the laboratory instructor. However, two points of safety deserve attention. First, a properly adjusted flame can be difficult to see: exercise care when working around a lit burner. If you have long hair, tie it back or put it up. Secondly, the porcelain crucible and top will become extremely hot (upwards of 600°C!) during the heating phases of the experiment. Consequently, manipulation of the crucible and lid requires the use of crucible tongs. These issues will be addressed by the lab instructor.

![Figure 3.1](image_url)

The experiment involves three heating and cooling cycles. The cooling process is best accomplished by allowing the crucible to cool for 3-4 minutes on the ring stand after the flame has been turned off. After this initial cooling period, the crucible and top are transferred using crucible tongs, to the wire gauze; do not place the lid on the crucible when cooling. The crucible should be allowed an additional 5-7 minutes of cooling on the gauze.

The use of the balance was discussed in the first experiment. In the current experiment, all mass determinations are performed by measuring the mass of the crucible and its contents. It is important that the crucible be thoroughly dried before measuring its mass. Consequently, the first heating and cooling cycle serves only to evaporate any residual water present in the crucible. Since the mass of the dried crucible will be subtracted from each subsequent measurement, it is vital for a successful experiment that all three places to the right of the decimal be recorded.
**Equipment**
porcelain crucible and lid, crucible tongs, clay triangle support, wire gauze, ring stand and ring, Bunsen burner

**Chemicals**
magnesium ribbon, water

**Procedure**
1. Inspect the crucible and report any cracks to the lab instructor. Place the empty crucible and lid (the lid should be ajar) upon the clay triangle support as shown in Figure 3.1.
2. Heat the crucible for 5 minutes at full heat (the bottom of the crucible should sit on top on the inner cone of flame). After 5 minutes of heating, cool the crucible as described in the procedural notes.
3. Coil the magnesium ribbon provided into a disc approximately 3/4 of an inch in diameter. **Do not coil too tightly!** It is important that the surface of the magnesium be exposed to the air.
4. Check to make sure the crucible is sufficiently cool to handle. Measure the mass of the empty crucible and lid. Record this mass in the data table. Place the coil of magnesium ribbon in the bottom of the crucible and record the mass of the crucible, lid and magnesium in the data table. **USE ALL DIGITS SHOWN ON THE BALANCE!**
5. Heat the crucible, lid and magnesium for 10 minutes at full heat. It is important that the lid is ajar to ensure access to oxygen.
6. Cool the crucible, lid and contents in the manner previously described. While cooling, heat approximately 30 mL of water in a beaker to the point of steaming but not boiling. When the crucible is cooled sufficiently, add 20 drops of the hot water to the crucible. Carefully note the odor of ammonia after this addition.
7. Place the crucible with the lid slightly ajar upon the clay triangle support and heat gently for 5 minutes. Gentle heating is accomplished by setting the bottom of the crucible at the top of the outer flame. Heat for an additional five minutes at high heat and then turn off the burner and allow the crucible to cool.
8. Once the crucible is cool to touch, record the mass of the crucible, lid and magnesium oxide in the data table.
9. Ask the laboratory instructor to check the crucible contents and data table entries. Complete question 1 before asking the instructor/grader to check your work.
10. After the crucible and contents have been inspected by the instructor, the magnesium oxide should be deposited in the plastic tub and wiped out with a paper towel.
11. Use the recorded data to answer the remaining questions in the conclusion and discussion section.
Data Table

<table>
<thead>
<tr>
<th></th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>mass of dry, empty crucible and lid (measured, grams)</td>
</tr>
<tr>
<td>II</td>
<td>mass of magnesium, crucible and lid (measured, grams)</td>
</tr>
<tr>
<td>III</td>
<td>mass of magnesium oxide, crucible and lid (measured, grams)</td>
</tr>
<tr>
<td>IV</td>
<td>mass of magnesium reacted (calculated: II - I = IV)</td>
</tr>
<tr>
<td>V</td>
<td>mass of magnesium oxide formed (calculated: III - I = V)</td>
</tr>
<tr>
<td>VI</td>
<td>mass of oxygen reacted (calculated: III - II = VI or V - IV = VI)</td>
</tr>
</tbody>
</table>

Conclusion and Discussion

Be sure to copy down the equations provided by the instructor during the prelab.

1. Calculate the percentage of oxygen in magnesium oxide.

2. Calculate the relative atomic mass of magnesium using the method described by the lab instructor. Assume that there are two magnesium atoms for each oxygen atom and that an oxygen atom has a mass of 16.0 amu.

3. Repeat the calculation from question 2 assuming one magnesium atom for every oxygen atom; continue using 16.0 amu for an oxygen atom. Repeat the calculation assuming 2 oxygen atoms for each magnesium atom. Refer to the periodic table in the lab to determine which combining ratio is correct.

4. There is always error involved in experimental results. Determine the percentage error in your answer for magnesium using the formula:
   
   \[ 100\% \times \frac{\text{known value} - \text{experimental value}}{\text{known value}} \]