

1. Stoichiometric Determinations

Objective

Stoichiometric measurements are among the most important in chemistry, indicating the proportions by mass in which various substances react. In this experiment, three examples of stoichiometric determinations will be investigated. The reactions to be studied are between two common acids (HCl, H₂SO₄) and sodium hydroxide (Choice I), sodium hydrogen carbonate and hydrochloric acid (Choice II), and magnesium and molecular oxygen, O₂ (Choice III).

Choice I. Stoichiometry and Limiting Reactant

Introduction

The concept of a limiting reactant is very important in the study of the stoichiometry of chemical reactions. The **limiting reactant** is the reactant that controls the amount of product possible for a process because once the limiting reactant has been consumed, no further reaction can occur.

Consider the following balanced chemical equation:



Suppose we set up a reaction in which we combine 1 mol of substance A and 1 mol of substance B. From the stoichiometry of the reaction, and from the amounts of A and B used, we would predict that exactly 1 mol of product C should form.

Suppose we set up a second experiment involving the same reaction, in which we again use 1 mol of A, but this time we only use 0.5 mol of B. Clearly, 1 mol of product C will *not* form. According to the balanced chemical equation, substances A and B react in a 1:1 ratio, and with only 0.5 mol of substance B, there is not enough substance B present to react with the entire 1 mol of substance A. Substance B would be the *limiting reactant* in this experiment. There would be 0.5 mol of unreacted substance A remaining once the reaction had been completed, and only 0.5 mol of product C would be formed.

Suppose we set up a third experiment, in which we combine 1 mol of substance A with 2 mols of substance B. In this case, substance A would be the limiting reactant (which would control how much product C is formed), and there would be an excess of substance B present once the reaction was complete.

One way of determining the extent of reaction for a chemical process is to monitor the *temperature* of the reaction system. Chemical reactions nearly always either absorb or liberate heat energy as they occur, and the amount of heat energy transferred will be *directly proportional* to how much product has formed. In this experiment, you will monitor temperature changes as an indication of the extent of reaction using a simple thermometer.

In this experiment, you will prepare solutions of hydrochloric acid and sulfuric acid, and will determine the stoichiometric ratio in which these acids react with the base sodium hydroxide. You will perform several trials of these acid/base reactions, in which the amount of each reagent used is systematically varied between the trials. By monitoring the temperature changes that take place as the reaction occurs, you will have an index of the extent of reaction. The maximum extent of reaction will occur when the reactants have been mixed together in the correct stoichiometric ratio for reaction. If the reactants for a particular trial are *not* in the correct stoichiometric ratio, then one of these reactants will *limit* the extent of reaction, and will also limit the temperature increase observed during the experiment.

SAFETY PRECAUTIONS

- **Wear safety glasses at all times while in the laboratory.**
- **The acid and base solutions used in the experiment may be damaging to skin, especially if concentrated through evaporation of the water. Wash after handling. If spilled on the bench, consult with the instructor about clean-up.**

Apparatus/Reagents Required

3.0 M solutions of HCl, H₂SO₄, and NaOH; plastic foam cup; thermometer

Procedure

Record all data and observations directly on the report pages in ink.

Check your pre-laboratory calculations with the instructor before preparing the solutions needed for this experiment.

A. *Reaction Between HCl and NaOH*

When preparing dilute acid/base solutions, slowly add the more concentrated stock solution to the appropriate amount of water with stirring.

Using 600-mL beakers for storage, prepare 500. mL each of 1.0 M NaOH and 1.0 M HCl, using the stock 3.0 M solutions available. Be sure to measure the amounts of concentrated acid or base and water required with a graduated cylinder.

Stir the solutions vigorously with a stirring rod for one minute to mix. Keep the solutions covered with a watch glass when not in use.

Frequently, diluting a concentrated solution with water will result in a temperature increase. *Allow the solutions to stand for 5–10 minutes until they have come to the same temperature* (within $\pm 0.2^{\circ}\text{C}$). Be sure to rinse and wipe the thermometer before switching between solutions.

Obtain a plastic foam cup for use as a reaction vessel: an insulated cup is used so that the heat liberated by the reactions will not be lost to the room during the time frame of the experiment.

Using a graduated cylinder, measure 45 mL of 1.0 M HCl. Pour it into the plastic cup and determine the temperature of the HCl (to the nearest 0.2°C). Record this temperature on your report sheet.

Measure 5.0 mL of 1.0 M NaOH in a 10-mL graduated cylinder.

Add the NaOH to the HCl in the plastic cup all at once and *carefully* stir the mixture with the thermometer. Determine and record the *highest temperature reached* as the reaction occurs.

Rinse and dry the plastic cup.

Perform the additional reaction trials indicated in Table I on the report sheet. In each case, measure the amounts of each solution carefully with a graduated cylinder. For each trial, record the initial temperature of the solution in the plastic cup, as well as the highest temperature reached during the reaction.

B. Reaction Between H₂SO₄ and NaOH

When preparing dilute acid/base solutions, slowly add the more concentrated stock solution to the appropriate amount of water with stirring.

Using 600-mL beakers for storage, prepare 500. mL each of 1.0 M NaOH and 1.0 M H₂SO₄, using the stock 3.0 M solutions available. Be sure to measure the amounts of concentrated acid or base and water required with a graduated cylinder.

Stir the solutions vigorously with a stirring rod for one minute to mix. Keep the solutions covered with a watch glass when not in use.

Allow the solutions to stand for 5–10 minutes until they have come to the same temperature (within ±0.2°C). Be sure to rinse and wipe the thermometer before switching between solutions.

Using the procedure described earlier, perform the reaction trials indicated in Table II on the report sheet for H₂SO₄ and NaOH. In each case, measure the amounts of each solution carefully with a graduated cylinder. For each trial, record the initial temperature of the solution in the plastic cup, as well as the highest temperature reached during the reaction.

Interpretation of Results

Based on the volume and concentration of the solutions used in each experiment, calculate the number of moles of each reactant used in each trial. For example, if you used 25.0 mL of 3.0 M HCl solution, the number of moles of HCl present would be

$$25.0 \text{ mL} \times \frac{1 \text{ L}}{1000 \text{ mL}} \times 3.0 \text{ M} = 0.075 \text{ mol}$$

Record these values in Tables I and II on the report sheet as appropriate.

To most clearly demonstrate how the extent of reaction in each of the experiments is determined by the limiting reactant, prepare two **graphs** of your

experimental data. One graph should be for the reaction between HCl and NaOH; the second graph should be for the reaction between H₂SO₄ and NaOH.

Set up your graphs so that the vertical axis represents the *temperature change* measured for a given trial. Set up the horizontal axis to represent the *number of moles of NaOH* used in the trial. Plot each of the data points with a sharp pencil.

You should notice that each of your graphs consists of a set of *ascending* points and a set of *descending* points. For each of these sets of points, use a ruler to draw the best straight line through the points.

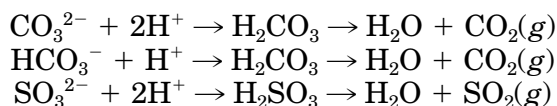
The **intersection** of the lines through the ascending points and the descending points in each graph represents the maximum extent of reaction for the particular experiment. From the maximum point on your graph, draw a straight line down to the horizontal axis. Read off the horizontal axis the number of moles of NaOH (and acid) that have reacted at this point. According to your graph, in what stoichiometric ratio do HCl and NaOH react? According to your graph, in what stoichiometric ratio do H₂SO₄ and NaOH react?

For each of your graphs, indicate which points represent the acid as limiting reactant, and which points represent NaOH as limiting reactant. Staple your two graphs to the report pages.

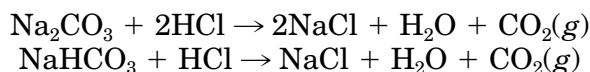
Choice II. Stoichiometry of a Gas Evolution/Neutralization Reaction

Introduction

Several common anions, when acidified, evolve *gases*. The net ionic reactions of the carbonate, hydrogen carbonate, and sulfite ions are examples of this:



Now consider the reactions



These reactions are basically those given in the first two of the preceding net ionic reactions. Since one of the products of the reaction is a gas (CO₂), and a second of the products can be conveniently vaporized by heating (H₂O), the stoichiometry of the reaction can be studied by collecting and weighing the third product of the reaction (NaCl). A similar study could be made of the reaction of the sulfite ion, but since the product gas SO₂ is toxic and noxious, this will not be done in this experiment.

In this experiment, you will treat weighed samples of sodium carbonate and sodium hydrogen carbonate dropwise with dilute hydrochloric acid until the reactions are complete. You will then evaporate the water from the samples. The quantity of sodium chloride produced in each reaction will be determined

and compared to the theoretical amount of NaCl that should have been produced from the stoichiometric ratios of the balanced chemical equations for the reactions.

The carbonate ion and the hydrogen carbonate ion are both bases in aqueous solution, and the reactions outlined earlier are typical acid/base reactions, in which one of the products of the reaction happens to be a *gas* under ordinary laboratory conditions. As in many acid/base reactions, an indicator—a dye that is sensitive to changes in pH—will be used to signal when the reactions are complete.

SAFETY PRECAUTIONS

- **Wear safety glasses at all times while in the laboratory.**
- **Hydrochloric and sulfuric acids are damaging to the skin and vapors of HCl are toxic. Keep the HCl in the exhaust hood.**
- **Considerable frothing will result as hydrochloric acid is added to the samples. Keep the casserole covered with a watch glass during addition of the acid. Beware of spattering.**
- **Evaporation of water from the samples must be done only over a boiling water bath or under a heating lamp. Dangerous spattering of hot salts will result if the evaporation is done over an open flame.**

Apparatus/Reagents Required

Porcelain or glass evaporating dish, sodium carbonate, sodium bicarbonate, 3.0 M hydrochloric acid, Pasteur pipet/bulb, methyl red indicator

Procedure

Clean a 50–60-mm-diameter casserole dish with soap and water. If any solid material in the casserole cannot be removed with simple washing, consult with the instructor about other methods for cleaning. Rinse the casserole with distilled water and wipe dry with a towel.

On a wire gauze on a ringstand, heat the casserole on a low flame for 5 minutes to dry it. Move the flame occasionally during the heating so that all portions of the casserole are heated. Allow the casserole to cool completely to room temperature.

Weigh the casserole to at least the nearest 0.01 g and record. Reheat the casserole on the wire gauze for an additional 5 minutes and reweigh after cooling completely. If the second mass determined for the casserole differs from the first mass by more than 0.02 g, reheat and reweigh until constant mass is achieved (within 0.02 g).

Add about half a teaspoon of sodium carbonate, Na₂CO₃, to the casserole and reweigh (to the nearest 0.01 g). Record. Calculate the mass of sodium carbonate taken.

Moisten the sodium carbonate with 4–5 mL of distilled water and add 2 drops of methyl red indicator (the mixture will be yellow). Cover the casserole with a watch glass to catch any material that may spatter. Obtain about 25 mL of 3.0 *M* hydrochloric acid in a clean beaker.

Based on the mass of sodium carbonate taken, calculate the volume of 3.0 *M* hydrochloric acid that should be required to react with the sodium carbonate.

Transfer the calculated volume of 3.0 *M* hydrochloric acid from the beaker to a graduated cylinder.

When adding HCl to the sample in the casserole, use a medicine dropper, and add the HCl down the pouring spout of the casserole without removing the watch glass. The sodium carbonate will froth and fizz as carbon dioxide is generated, and the watch glass will prevent loss of solid. Begin adding 3.0 *M* hydrochloric acid dropwise to the casserole from the portion measured in the graduated cylinder.

Continue adding HCl with the dropper from the graduated cylinder until there is approximately 1 mL remaining in the graduate. During the initial addition of the HCl, the indicator may change to red. This may *not* signal completion of the reaction, however, because some carbon dioxide may remain in solution at this point, thereby affecting the pH of the mixture.

Transfer the casserole to the wire gauze/ringstand and heat with a low flame until the mixture *just begins to boil*. This heating is only to drive off carbon dioxide: do *not* attempt to boil off the water from the mixture at this point. As carbon dioxide is evolved on heating, the mixture should turn yellow again.

Add additional HCl dropwise from the graduated cylinder until the mixture in the casserole turns a permanent pale red.

Use a stream of distilled water from a plastic wash bottle to rinse any solids that may have collected on the bottom of the watch glass into the casserole.

Set up a 400- or 600-mL beaker that can accommodate the casserole on the wire gauze/ringstand, add about 300 mL of tap water, and bring the water to boiling to provide a steam bath.

Place the casserole in the mouth of the beaker of boiling water and begin heating to evaporate water. The evaporation of water will take a considerable amount of time, so go on to any other work that may have been assigned. Replace the water in the beaker as needed to maintain the steam bath.

Do not attempt to hurry the evaporation of water by using a direct flame on the casserole. As the mixture in the casserole becomes more concentrated by evaporation, it will tend to “bump” and spatter badly if directly heated, leading to loss of product. Spattering of hot solids is also dangerous.

After the *solid* (NaCl) in the casserole is almost completely dry, remove the casserole from the steam bath. Begin heating the casserole directly with a *very small* flame.

If the casserole contents begin to spatter, not enough water has been evaporated and the casserole should be returned to the steam bath for additional

slow heating. If no spattering occurs, continue heating the casserole directly with a small flame for 5 minutes.

Increase the size of the flame somewhat, and continue heating the casserole for an additional 5-minute period to remove all moisture. Let the casserole cool completely to room temperature.

When the casserole has cooled completely, determine the mass of the casserole and contents (to the nearest 0.01 g). Calculate the mass of sodium chloride in the casserole.

Based on the mass of sodium carbonate taken originally, calculate the mass of sodium chloride that should in theory be produced by the reaction. Based on the actual yield of NaCl and the calculated theoretical yield, calculate the percent yield for your experiment.

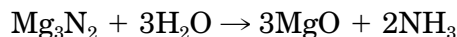
If time permits and your instructor so directs, repeat the experiment, substituting sodium hydrogen carbonate (bicarbonate) for the sodium carbonate.

Choice III. Determination of a Formula

Introduction

Magnesium metal is a moderately reactive elementary substance. At room temperature, magnesium reacts only very slowly with oxygen and can be kept for long periods of time without appreciable oxide buildup. At elevated temperatures, however, magnesium will ignite in an excess of oxygen gas, burning with an intensely white flame and producing magnesium oxide. Because of the brightness of its flame, magnesium is used in flares and in photographic flashbulbs.

In this experiment, however, you will be heating magnesium in a closed container called a **crucible**, exposing it only gradually to the oxygen of the air. Under these conditions, the magnesium will undergo a more controlled oxidation, gradually turning from shiny metal to grayish-white powdered oxide. Because the air also contains a great deal of nitrogen gas, a portion of the magnesium being heated may be converted to magnesium nitride, Mg_3N_2 , rather than magnesium oxide. Magnesium nitride will react with water and, on careful heating, is converted into magnesium oxide



The ammonia produced by this reaction can be detected by its odor, which is released on heating the mixture.

Magnesium is a Group IIA metal, and its oxide should have the formula MgO . Based on this formula, magnesium oxide should consist of approximately 60% magnesium by weight. By comparing the weight of magnesium reacted, and the weight of magnesium oxide that results from the reaction, this will be confirmed.

SAFETY PRECAUTIONS

- **Wear safety glasses at all times while in the laboratory.**
- **Magnesium produces an intensely white flame if ignited, which may be damaging to the eyes. If the magnesium used in this experiment ignites in the crucible by accident, *immediately* cover the crucible and stop heating. Do not look directly at magnesium while it is burning.**
- **When water is added to the crucible to convert magnesium nitride to magnesium oxide, the contents of the crucible may spatter when heated. Use only gentle heating in evaporating the water. Do not heat the crucible strongly until nearly all the water has been removed.**
- **Use crucible tongs to handle the hot crucible and cover.**
- **Hydrochloric acid is damaging to skin and clothing. If it is spilled, wash immediately and inform the instructor.**

Apparatus/Reagents Required

Porcelain crucible and cover, crucible tongs, clay triangle, magnesium turnings (or ribbon), pH paper, 6 M HCl

Procedure

Record all data and observations directly in your notebook in ink.

Obtain a crucible and cover and examine. The crucible and cover are extremely fragile and expensive. Use caution in handling them.

If there is any loose dirt in the crucible, moisten and rub it gently with a paper towel to remove the dirt. If dirt remains in the crucible, bring it to the hood, add 5–10 mL of 6 M HCl and allow the crucible to stand for 5 minutes. Discard the HCl and rinse the crucible with water. If the crucible is not clean at this point, consult with the instructor about other cleaning techniques, or replace the crucible. After the crucible has been cleaned, use tongs to handle the crucible and cover.

Set up a clay triangle on a ringstand. Transfer the crucible and cover to the triangle. The crucible should sit firmly in the triangle (the triangle's arms can be bent slightly if necessary).

Begin heating the crucible and cover with a small flame to dry them. When the crucible and cover show no visible droplets of moisture, increase the flame to full intensity, and heat the crucible and cover for 5 minutes.

Remove the flame, and allow the crucible and cover to cool to room temperature.

When the crucible and cover are completely cool, use tongs to move them to a clean dry watch glass or flat glass plate. Do not place the crucible directly on the lab bench. Weigh the crucible and cover to the nearest milligram (0.001 g).

Return the crucible and cover to the clay triangle. Reheat in the full heat of the burner flame for 5 minutes. Allow the crucible/cover to cool completely to room temperature.

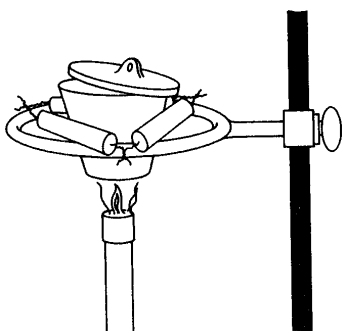
Reweigh the crucible after it has cooled. If the weight this time differs from the earlier weight by more than 5 mg (0.005 g), reheat the crucible for an additional 5 minutes and reweigh when cool. Continue the heating/weighing until the weight of the crucible and cover is constant to within 5 mg.

Add approximately 1 teaspoon of magnesium turnings (or about 8 inches of magnesium ribbon coiled into a spiral) to the crucible.

Using tongs, transfer the crucible/cover and magnesium to the balance and weigh to the nearest mg (0.001 g).

Set up the crucible on the clay triangle with the cover very slightly ajar. (See Figure 1-1.) With a very small flame, begin heating the crucible gently.

FIGURE 1-1
Set-up for
oxidation of
magnesium.



If the crucible begins to smoke when heated, *immediately* cover the magnesium completely and remove the heat for 2–3 minutes. The smoke consists of the magnesium oxide product and must not be lost from the crucible.

Continue to heat gently for 5–10 minutes with the cover of the crucible slightly ajar. Remove the heat and allow the crucible to cool for 1–2 minutes.

Remove the cover and examine the contents of the crucible. If portions of the magnesium still demonstrate the shiny appearance of the free metal, return the cover and heat with a small flame for an additional 5 minutes; then re-examine the metal. Continue heating with a small flame until no shiny metallic pieces are visible.

When the shiny magnesium metal appears to have been converted fully to the dull gray oxide, return the cover to its slightly ajar position, and heat the crucible with the full heat of the burner flame for 5 minutes. Then slide the cover to about the half-open position and heat the crucible in the full heat of the burner flame for an additional 5 minutes.

Remove the heat and allow the crucible and contents to cool completely to room temperature. Remove the crucible from the clay triangle and set it on a sheet of clean paper on the lab bench.

With a stirring rod, gently break up any large chunks of solid in the crucible. Rinse any material that adheres to the stirring rod into the crucible with a few drops of distilled water. With a dropper, add about 10 drops of distilled water to the crucible, spreading the water evenly throughout the solid.

Return the crucible to the clay triangle, and set the cover in the slightly ajar position. With a very small flame, begin heating the crucible to drive off the water that has been added. Beware of spattering during the heating. Remove the flame and close the cover of the crucible if spattering occurs.

As the water is driven off, hold a piece of moistened pH paper (with forceps) in the stream of steam being expelled from the crucible. Any nitrogen that had reacted with the magnesium is driven off as ammonia during the heating and should give a basic response with pH paper (you may also note the odor of ammonia).

When it is certain that all the water has been driven off, slide the cover so that it is in approximately the half-open position, and increase the size of the flame. Heat the crucible and contents in the full heat of the burner for 5 minutes.

Allow the crucible and contents to cool completely to room temperature. When completely cool, weigh the crucible and contents to the nearest milligram (0.001 g).

Return the crucible to the triangle and heat for another 5 minutes in the full heat of the burner flame. Allow the crucible to cool completely to room temperature and reweigh. The two measurements of the crucible and contents should give weights that agree within 5 mg (0.005 g). If this agreement is not obtained, heat the crucible for additional 5-minute periods until two successive weighings agree within 5 mg.

Clean out the crucible, and repeat the determination.

Calculate the weight of magnesium that was taken, as well as the weight of magnesium oxide that was present after the completion of the reaction. Calculate the percentage of magnesium in the magnesium oxide from your experimental data. Calculate the mean for your two determinations.

Calculate the theoretical percentage of magnesium (by mass) in magnesium oxide, and compare this to the mean experimental value. Calculate the percent error in your determination.

Stoichiometric Determinations

Date: Student name:
Course: Team members:
Section:
Instructor:

Prelaboratory Questions

Choice I. Stoichiometry and Limiting Reactant

1. Magnesium metal reacts with chlorine gas to produce magnesium chloride, MgCl_2 .
 - a. Write the balanced chemical equation for the reaction.

 - b. If 5.00 g magnesium is combined with 10.0 g of chlorine, show by calculation which substance is the limiting reactant, and calculate the theoretical yield of magnesium chloride for the reaction.

2. How many mL of 3.0 M HCl are required to prepare 500. mL of 1.0 M HCl solution? Show the method of calculation clearly.

Stoichiometric Determinations

Date: Student name:
Course: Team members:
Section:
Instructor:

Results/Observations

Choice I. Stoichiometry and Limiting Reactant

A. Reaction Between Hydrochloric Acid and Sodium Hydroxide

Table I

mL 1.0 M HCl	Moles HCl	mL 1.0 M NaOH	Moles NaOH	Temperature change, °C
45.0	5.0
40.0	10.0
35.0	15.0
30.0	20.0
25.0	25.0
20.0	30.0
15.0	35.0
10.0	40.0
5.0	45.0

Intersection of ascending and descending portions of graph, mol NaOH

Stoichiometric ratio, mol HCl/mol NaOH at intersection

Balanced chemical equation for the reaction between HCl and NaOH

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B. Reaction Between Sulfuric Acid and Sodium Hydroxide

Table II

mL 1.0 M H ₂ SO ₄	Moles H ₂ SO ₄	mL 1.0 M NaOH	Moles NaOH	Temperature change, °C
45.0	5.0
40.0	10.0
35.0	15.0
30.0	20.0
25.0	25.0
20.0	30.0
15.0	35.0
10.0	40.0
5.0	45.0

Intersection of ascending and descending portions of graph, mol NaOH

Stoichiometric ratio, mol H₂SO₄/mol NaOH at intersection

Balanced chemical equation for the reaction between H₂SO₄ and NaOH

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Questions

1. Why was it important to wait until the solutions to be mixed had come to the same temperature?

2. Suppose a similar experiment had been done using 1.0 M H₃PO₄ solution as the acid. What would be the stoichiometric ratio, mol H₃PO₄/mol NaOH, at the intersection of the ascending and descending portions of the graph for such an experiment? Explain.

Stoichiometric Determinations

Date: Student name:
Course: Team members:
Section:
Instructor:

Results/Observations

Choice II. Stoichiometry of a Gas Evolution/Neutralization Reaction

Reaction of Sodium Carbonate

Mass of casserole after first heating, g

Mass of casserole after second heating, g

Mass of casserole plus sodium carbonate, g

Mass of sodium carbonate taken, g

Approximate volume of 3 M HCl required, mL

Mass of casserole and sodium chloride, g

Mass of sodium chloride formed, g

Theoretical yield of sodium chloride, g

Percentage yield of sodium chloride

Reaction of Sodium Hydrogen Carbonate

Mass of casserole after first heating, g

Mass of casserole after second heating, g

Mass of casserole plus sodium carbonate, g

Mass of sodium carbonate taken, g

Approximate volume of 3 M HCl required, mL

Mass of casserole and sodium chloride, g

Mass of sodium chloride formed, g

Theoretical yield of sodium chloride, g

Percentage yield of sodium chloride

Stoichiometric Determinations

Date: Student name:
Course: Team members:
Section:
Instructor:

Results/Observations

Choice III. Determination of a Formula

	Trial 1	Trial 2
Weight of empty crucible (after first heating)
Weight of empty crucible (after second heating)
Weight of crucible with Mg
Weight of Mg taken
Weight of crucible/MgO (after first heating)
Weight of crucible/MgO (after second heating)
Weight of MgO produced
Weight of oxygen gained
% magnesium in the oxide
Mean % magnesium	
Error %	

