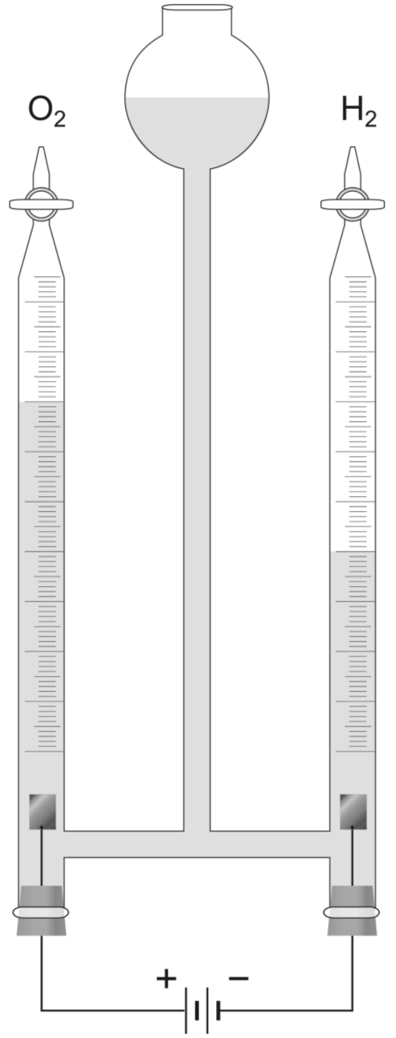
LAB MANUAL



SOPHOMORE NATURAL SCIENCE

THOMAS AQUINAS COLLEGE
2017

TABLE OF CONTENTS
SOPHOMORE LAB MANUAL

Laboratory Regulations

Students are responsible for following all of the regulations below. Failure to follow any of these regulations may be a serious disciplinary matter.

a. Use of Laboratory

Students may not use the laboratory for any purpose without the express consent of the laboratory tutor or lab assistatnt.

b. Safety in the Laboratory

It is the duty of the tutor to explain and promote safe procedures for doing experiments, and to oversee students when they are working the lab. The tutor will show you the location of the nearest fire extinguisher and explain the operation of the safety shower in the laboratory itself. There is a First Aid kit in the laboratory. Safety goggles **must** be worn **whenever** you are in the laboratory unless you are told otherwise by the tutor or laboratory assistant.

Safety Notices and Safety Equipment:

Pay careful attention to the safety notices in the lab manual and be sure to follow any instructions regarding safety equipment such as the use of gloves, safety glasses (or goggles), or lab aprons.

Injuries:

- If any chemical splashes on your face, especially into your eyes, go at once to the green eye-wash sink and rinse off your face and eyes with **copious** amounts of water from the spray. Then report to the tutor.
- If you receive a burn, immediately rinse the burn with cold water. Then report to the tutor.
- If you receive any other inuruies in the lab, no matter how slight, report it to the tutor.

Using the Bunsen Burner

- If you are unable to light the Bunsen burner, and you have had the burner gas on for more than 10 seconds, shut off the gas for at least 20 seconds before making another attempt to light the burner.
- When suspending a beaker over a Bunsen burner, double check to make sure that the ring stand is securely attached by tightening all thumb screws.
- Never leave a lighted burner unattended.
- When you turn of the burner gas, double check to make sure that the gas has been completely shut off.

Handling Chemicals

- Do not mix any lab chemicals other than those called for in the *Lab Manual* unless you are instructed to do so by a tutor or laboratory supervisor.
- Never place a container directly under your nose to smell its contents. Instead, use one hand to gently fan some of the odor towards your face.

- Chemical spills should be reported to the tutor, especially when dealing with corrosive, hazardous, or highly reactive chemicals (i.e. Sulfuric acid, Mercury, calcium)

Food and Drink

- Do not bring food or drinks into the lab.
- Wash your hands after each lab.

d. Attire

- Although the classroom dress code applies, you should not wear your best clothes when doing experiments since you will be working with a number of corrosive substances.
- No opened-toe shoes, or shoes that can be easily slipped off, should be worn when doing experiments in the lab.
- Secure loose hair and clothing before working in the lab. Hair is flammable and can easily catch fire in Bunsen burner.
- Avoid wearing contact lenses in the lab since chemicals splashed into the eyes can get trapped between the lens and eye, making it difficult to wash out the chemical.
- Avoid wearing synthetic fingernails since they are made of extremely flammable polymers.

e. Neatness

- Keep your work station neat and clean at all times.
- Follow the **Clean Up** instructions in the *Lab Manual*.
- Do not pour used chemicals down the drain unless you are instructed to do so.
- Poisonous substances, or substances that are highly corrosive, or caustic, are usually discarded in specially marked waste containers.
- Discard used solids (e.g., iron powder, sulfur), disposable gloves, wooden splints, and other rubbish in the trash can.
- **Broken glass** should be discarded in specially marked trash containers.
- Clean your glassware at the end of each class and be sure to rinse with with **distilled water** using one of the wash bottles.

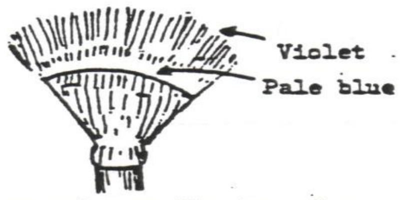
f. Use of Materials from Flasks and Bottles

- Always use a scoopula, or spatula, to transfer solid chemicals from storage containers. This is to ensure personal safety and to avoid contaminating lab chemicals.
- After taking liquid materials from flasks or storage bottles, **always** replace the stoppers (or lids), as many of these substances are volatile, and confusing stoppers (or lids) may introduce impurities.
- **Never** take the reagent bottles or flasks to your workstation, as they are for use by all.
- If by chance you take too much from a flask, discard the surplus in the waste jar at your station or on the back counter; never return anything to the flasks.

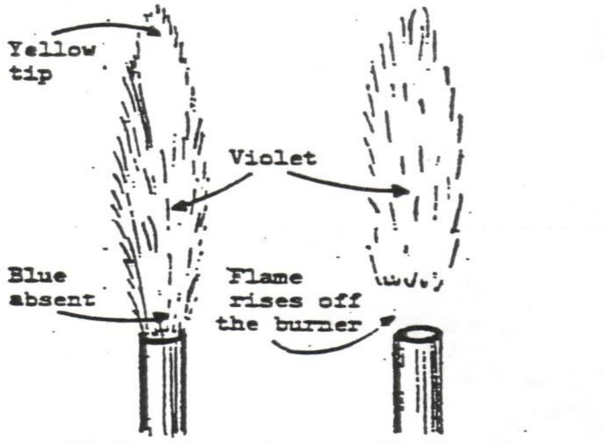
g. Behavior in General

The laboratory is a place of serious study, so you should treat the laboratory sessions with the same respect due the other classes. Some laboratory experiments can be dangerous, and all of them require careful movement and measurement. Thus, when in the laboratory a student must move slowly and deliberately. Fooling around, or the abuse of laboratory equipment, will not be tolerated in the laboratory.

h. Notes on the Use of the Burner:



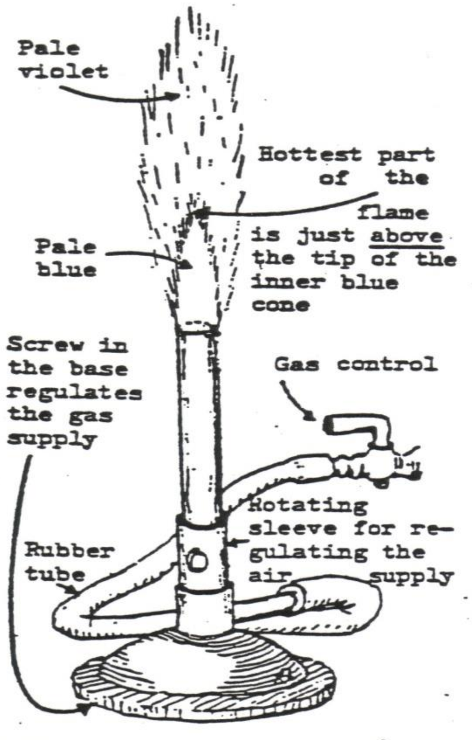
A flame spreader on the top of a Bunsen burner makes a flat flame like this



If the flame is like this, open the air regulator

If the flame is like this, turn down the gas

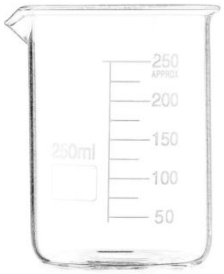
Figure 1



Bunsen burner with correct flame

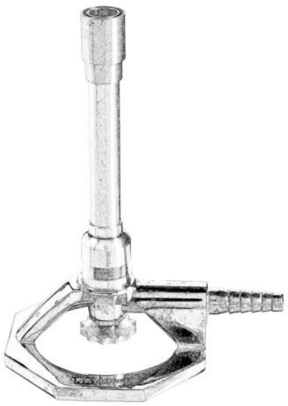
Glossary of Laboratory Equipment

Beaker – glass container used to mix, hold, or heat solutions and solid chemicals. Graduations do not measure volume with high accuracy.

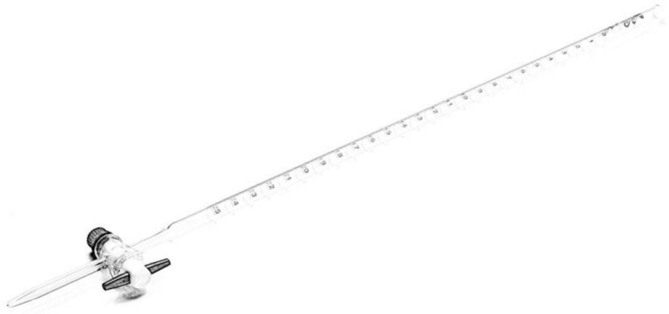


Beaker tongs – tongs with rubber coating to handle hot beakers.

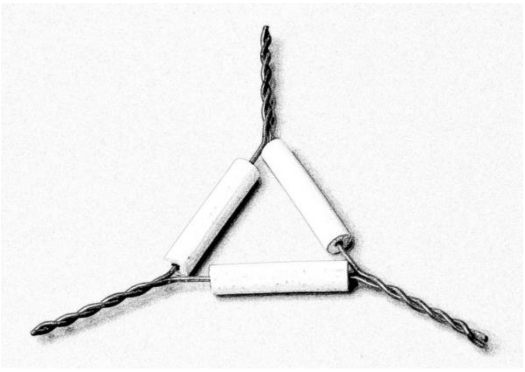
Bunsen burner – burner connected to gas outlet at its base with a rubber hose and producing a concentrated flame. Flame intensity and gas intake can be adjusted by turning a dial at the base.



Burette – used to deliver variable, measurable amounts of liquid drop by drop. Its inverted scale of volume graduations measures the volume delivered accurately. Opened by turning the stopcock at the bottom end to a position parallel to the burette.

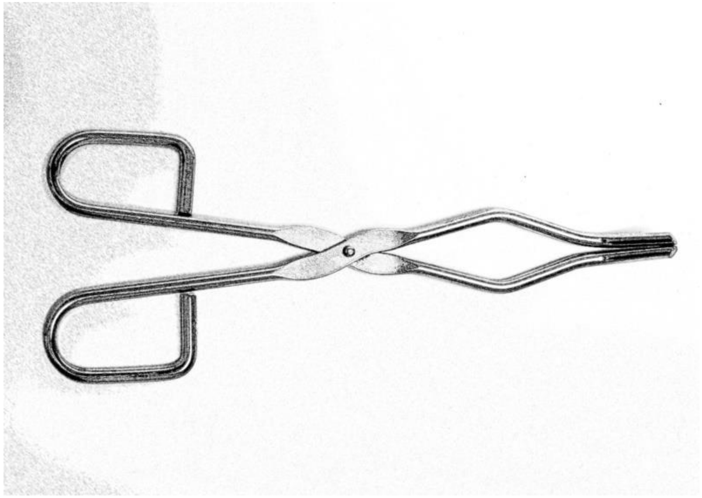


Clay triangle – used to support crucibles over Bunsen burner. Placed on top of a metal ring fixed to a ring stand.



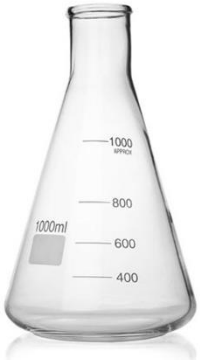
Crucible – heat-resistant ceramic dish used to melt or burn solid chemicals over a burner. Cover is used to contain gases being emitted.

Crucible tongs – metal tongs used to handle hot crucibles and other small items.



Disposable pipette – plastic pipette used to transfer small volumes of liquid.

Erlenmeyer flask – flat bottomed flask which, like a beaker, can be used to mix, dissolve chemicals into solutions, and heat or cool solutions. Narrow neck prevents spills and splashes during stirring or heating. Graduations do not measure volume with high accuracy.



Evaporating dish – ceramic dish used for separating liquids and solids from solution by heating over a burner; the solid collects on the surface of the dish after liquid is evaporated off.

Funnel, powder – short-stem funnel with wide neck used to transfer powder.

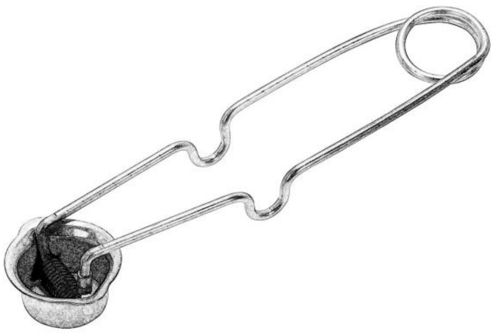
Funnel, filtering – long-stem funnel with a narrow neck used to transfer liquids. May be used with filter paper to separate components of a solution.

Mortar and pestle – used to grind solids into fine powders to speed the mixing process.

Scoopula – a spatula-like scoop utensil used to transfer solids to a weigh paper for weighing, to scrape solids from the sides of a crucible or test tube, and to cut soft materials.

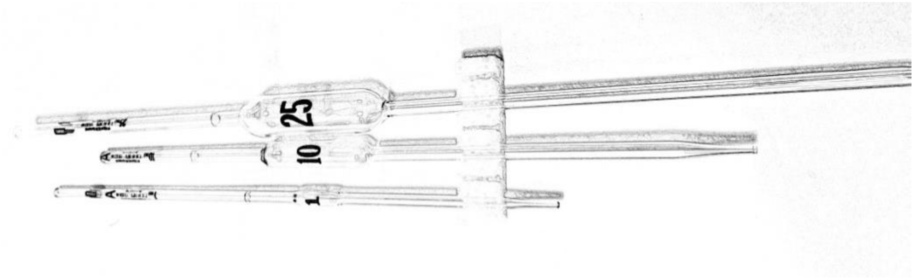
Stir rod and rubber policeman – glass rod used to manually stir solutions; assist in pouring liquids; and to transfer a single drop of a solution. The rubber policeman is a rubber spatula that fits on the end of a glass stir and is used to remove precipitates.

Striker – used to light Bunsen burners.



Test tube holder – spring handle tongs used for handling hot test tubes.

Volumetric pipette – used to deliver a fixed volume of liquid with very high accuracy. Marked with a single graduation line indicating single volume (i.e. 1 mL, 10 mL, 25 mL).

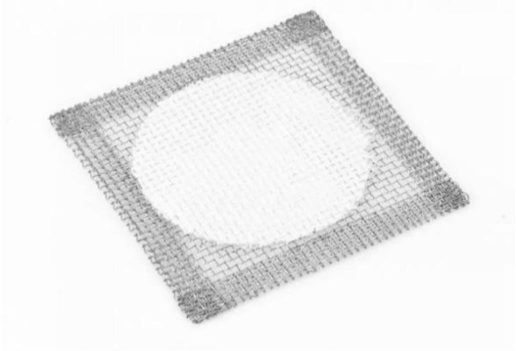


Wash bottle – plastic bottle fitted with a nozzle to spray solution in a small stream. Filled with distilled water and used to rinse lab glassware clean.

Watch Glass – used to cover beakers, evaporate water from solutions, or weigh out solid chemicals.

Weigh Boat and Weigh Paper – used to weigh solids on a lab scale.

Wire Gauze – thin sheet of wire mesh designed to spread the heat of a burner flame and to support beakers and other glassware heated over a burner.



Experiment 1: Classifying Substances

The principal aim of the Classifying Substances lab is to distinguish mixtures from compounds, and to identify those substances that might be simple or elemental substances. The experiment also introduces the student to some of the basic kinds of chemical changes that substances undergo and familiarizes them with basic laboratory techniques. It has two parts. In Part I you will employ some of the most common techniques used to identify and separate **mixtures**. In Part II you will observe several different kinds of chemical reactions that can be used to distinguish between mixtures and **compounds**, and between compounds and elements.

Part I: Mixtures

Synopsis

You are to separate four mixtures using the techniques described below, and identify the physical properties of the component substances that permit one to make the separation. The four mixtures you will attempt to separate are: (1) salt water, (2) a mixture of sand and salt, (3) a mixture of sand and iron filings, and (4) a mixture of sulfur and iron filings. Your tutor will demonstrate the separation of a fifth mixture, a 50/50 mixture of alcohol and water, by the process of distillation. If time permits, your tutor will also demonstrate the separation of a mixture of sulfur and iron filings using carbon disulfide, a poisonous and inflammable solvent.

Materials

Salt
Sand
Iron filings
Wash bottle w/distilled water
100 mL beakers (2)
Lab scale
Ring stand and ring
Wire gauze
Evaporating dish
Filter paper
Funnel
Magnet (and sandwich bag)
Scoopula

Before you attempt to separate the four mixtures, you will need to make them from the materials listed above by mixing the above materials in a small 100 mL beaker. You will only work with one mixture at a time so that your work station does not become overly crowded. Here are some rough guidelines for making your mixtures:

1) Salt water: dissolve about 1g of salt in about 10mL of distilled water
2) Sand and salt: mix about 1g of sand with about 1g of salt
3) Sand and iron: mix about 1g of sand with about 1g of iron filings
4) Sulfur and Iron: mix about .5g of sulfur with 1g of iron filings

Method

Below is a list of techniques you may use to separate the above mixtures. **Be sure to read the entire set of techniques before beginning.** You do not need to try every technique for each mixture. Use common sense.

Magnetic Separation

Magnetic separation is a technique used to separate substances that are attracted by a magnet from those that are not. When using the magnet to separate magnetic material, cover the end of the magnet with a sandwich bag. This will make it easy to separate the magnetic material from the end of the magnet (if you remove the end of the magnet from the plastic bag, the magnetic material will easily separate from the plastic).

Decantation

Decantation or decanting is a technique to separate a liquid and a solid, or two liquids, that have different densities. It consists in letting the substance with a greater density settle to the bottom and then pouring off the less dense liquid from the top.

Filtration

Filtration aids in separating solids suspended in a liquid by interposing a medium through which only the fluid can pass. The liquid which passes through the filter is called the “filtrate.” The solid material remaining is called the “residue.” Here is the procedure:

1. Place a funnel in the ring mounted on the ring stand and place a beaker under the funnel.
2. Take a circle of filter paper and fold it along the diameter. Make a second fold so that the paper is in quarters from its original shape. Place the quartered filter paper into the filter and open one layer of it so that it opens as a cone inside the funnel.
3. Examine the residue, if any, left on the filter paper. Does it look like one of the original constituents of the mixture?

Evaporation

Evaporation is a way of separating solid substances from a liquid solution. When a liquid is evaporated, assuming that the apparatus used is clean, a residue is sometimes left after complete evaporation. The residue shows that the original substance was a solution containing a liquid solvent (the liquid that evaporates) and the solute (the solid substance that remains after the solvent has evaporated). Evaporation can be accelerated by heating a small amount of the solution in an evaporation dish (or in small beaker). It is essential to make sure that any glassware used for evaporation be clean before you start the evaporation. Use distilled water to clean your evaporation dish or beaker (do not use tap water as it has minerals and impurities that will yield false results).

Selective Dissolution

Selective dissolution is a way to separate a mixture of two solid substances. If in a given solvent one substance is soluble and the other insoluble, then the two substances can be separated by selective dissolution followed by one of the other techniques outlined above (e.g., if one solid is dissolved by a solvent, then it can be separated from the other solid by decantation or filtration). The solvent you have to work with is distilled water.

Hint: Before attempting to use a solvent to separate a mixture, try testing whether the solvent will dissolve one of the individual components of your mixture before using it to separate the mixture. To test the solubility of a solid in a liquid take a small pinch of the solid and deposit the sample into a beaker with about 5 mL of liquid. Swirl the liquid in order to thoroughly mix the potential solvent and solute.

Results

Record your findings in the table below. Note which techniques were successful and which were not. Note the properties of the substances in the mixture that enabled you to separate the mixture. If you combined several techniques to separate a mixture, note your procedure.

Mixture	**Magnetic Separation**	**Decantation**	**Filtration**	**Evaporation**	**Selective Dissolution**	**Notes** - **Which properties were used to separate the mixture?** - **Was one technique combined with another (e.g., dissolution and decantation)?** - **Did you encounter any difficulties?**
Salt & Water						
Sand & Salt						
Sand & Iron						
Sulfur & Iron						

(In the appropriate box, write a "√" for a successful result.)

Cleanup

Clean your lab station before you leave. Salt water can be washed down the drain. Sand, iron filings, and sulfur should be thrown in the trash. Here is how: holding the beaker upside down over a trash can, rinse the beaker containing sand, iron filings, or sulfur with a wash bottle. **Do not put sand, iron filings, or sulfur down the drain!** Wash the funnel and beakers, dispose of any used filters, and wipe down your lab station.

Demonstration 1: Distillation of a Mixture of Alcohol and Water

Distillation is used to separate two liquids with different boiling points by selective evaporation and condensation. Your tutor will use this technique to demonstrate how to separate a mixture of alcohol and water.

Materials

Distilled water
Methyl alcohol (methanol)
Distillation setup (see diagram)
Collection cylinder labeled “methanol”
Collection cylinder labeled “distilled water”
Lighter
Crucible tongs
Cotton ball
Alcohol thermometer

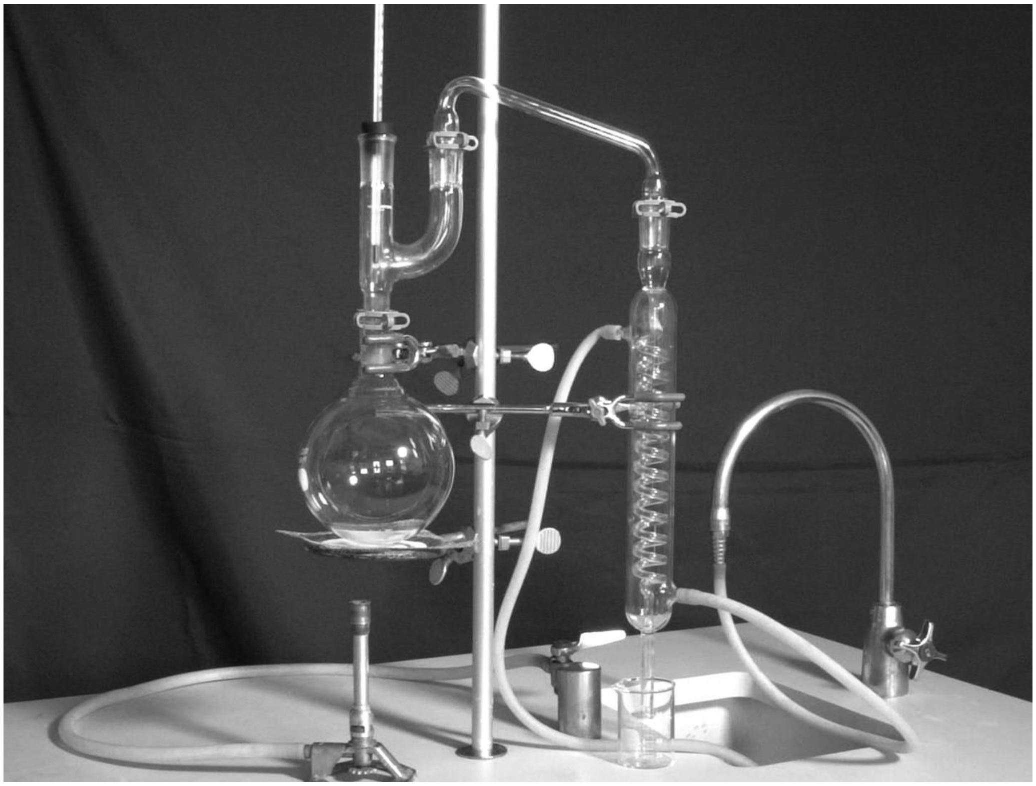
Safety Notice

Methyl alcohol is a volatile substance that is toxic, so avoid inhaling the fumes and protect your hands with nitrile gloves. It is also highly inflammable, so keep it away from an open flame.

This is the procedure:

1. Mix 20 mL of distilled water with 20 mL of methyl alcohol in a small beaker.
2. Add the mixture of alcohol and water into the distillation flask that sits on top of the wire gauze/ring stand. Add 5-6 boiling stones to the flask. Attach a Claisen adapter to the top of the flask. (A Claisen adapter is a piece of glassware that attaches directly to the top of the flask that has an opening to insert a thermometer and a sidearm used to attach a condenser). Note: vacuum grease should always be used when joining ground glass joints.
3. Using a thermometer adapter (or one hole stopper), insert the thermometer into the distillation head and adjust the tip so that it is the same height as the sidearm going to the condenser. The thermometer is used to monitor the vapor entering into the condenser.
4. Attach the condenser to the Claisen adapter. This may require an additional adapter. Note: a Graham condenser, which consists of coiled glass tube passing through an outer jacket of water, must be installed vertically. A Liebig condenser, which consists of a straight glass inner tube passing through an outer jacket of water, is typically installed horizontally.
5. Attach a piece of latex tubing from the distillation nozzle on the faucet to the water inlet on the bottom end of the condenser. Attach another piece of latex tubing to the water outlet on the upper side of the condenser and place the other end of this tube in the sink. Gradually turn on the faucet and check for leaks. The water should flow from the faucet into the water jacket on the condenser and then into the sink. The purpose of the water jacket is to cool the hot vapors coming from the distillation flask.
6. Have two beakers or collection cylinders that fit easily underneath the bottom of the condenser to catch the distillate.

The distillation setup using a Graham condenser should look something like this:



The distillation setup using a Liebig condenser should look something like this:

7. Light the Bunsen burner and begin heating the distillation flask.
8. The alcohol will begin to boil first because its boiling point is lower than that of water which is roughly 100°C. Once the liquid in the flask begins to boil, take note of the temperature.

Soon you will begin to see alcohol condense and begin to fill the collection cylinder.

9. When the first collection cylinder contains 20mL, note the temperature and replace the “methanol” collection cylinder with the “distilled water” collection cylinder. After all of the liquid in the flask has evaporated, turn off your burner.
10. You can test to see whether you have successfully separated alcohol and water by testing the flammability of the two liquids. Alcohol burns with a faint blue flame that can be hard to see, so it is best to dim the lights before proceeding. Saturate a cotton ball with what you believe to be alcohol and test whether it is flammable by holding it in the burner flame using crucible tongs. **Be careful: methyl alcohol is extremely flammable!** Perform the same test with a cotton ball saturated with what you believe to be water.

Cleanup

The distilled methanol can be reused. It should be poured into a plastic container labelled “reclaimed methanol” and stored in the lab fridge. **Do not pour methanol down the drain!**

Demonstration 2: Separating a Mixture of Iron and Sulfur with Carbon Disulfide

If time permits, you tutor will demonstrate how to separate a mixture of iron and sulfur using carbon disulfide. As you will likely discover, sulfur is insoluble in water, but it does dissolve in other solvents such as toluene, xylene, and especially in carbon disulfide. Since carbon disulfide produces a noxious gas and is extremely flammable, your tutor will perform this demonstration in the fume hood.

Materials

Sulfur
Iron filings
Carbon disulfide
Nitrile gloves (15 mil)
Lab scale
2 100 mL beaker
Scoopula
Disposable pipette
Watch glass (large)

Safety Notice

Carbon Disulfide is extremely toxic and highly flammable. Any experiments with carbon disulfide must be carried out in a well-ventilated fume hood. Do not under any circumstances expose this substance to heat or flame.

Here is the procedure:

1. For those students who did not try dissolving sulfur in water, begin by illustrating that a mixture of sulfur and iron cannot be separated using water as a solvent since sulfur is insoluble in water. To do this, take a pinch of sulfur and add it to a small beaker containing about 10mL of distilled water.

2. Mix about one gram of sulfur with two grams of iron filings in a 100 mL beaker, stirring the mixture with a scoopula.
3. Using a disposable pipette, add about 5-10 mL of carbon disulfide to the mixture and gently swirl the beaker to help the solvent dissolve the sulfur. **This must be performed under the fume hood! Carbon disulfide fumes are toxic.**
4. Once the sulfur is dissolved in the solvent, carefully decant the solution onto a large watch glass.
5. Add an additional 3-5 mL of carbon disulfide to dissolve any remaining sulfur and again decant the solution onto the watch glass.
6. As the carbon disulfide evaporates, you should observe the formation of yellow sulfur crystals on the watch glass. Carbon disulfide is easily evaporated at room temperature. **Do not under any circumstance expose carbon disulfide to heat! It is a volatile and toxic chemical and, when burnt, releases a toxic gas.**

Cleanup

Do not attempt to clean up the iron filings or sulfur crystals until the carbon disulfide has completely evaporated. Once the carbon disulfide has evaporated, the iron filings and sulfur can be thrown into the trash. **Do not put the iron filings or sulfur down the drain!** Clean out the iron filings from the beaker with a scoopula and a dry paper towel. Wipe off the sulfur crystals from the watch glass with a dry paper towel. The beaker and watch glass should then be washed thoroughly and dried. The disposable pipette should be thrown away.

Experiment 1: Classifying Substances (continued)

Part II: Compounds

Synopsis

The overall aim of the classifying substances lab is to distinguish mixtures from compounds, and to identify those substances that might be simple or elemental substances. Part II of the classifying substances lab focuses on ways of identifying **composite substances**, or **compounds**, those substances that are generated from a combination of more than one substance, or decompose into more than one substance. Identifying compound substances also helps—negatively, or indirectly—to discover or identify **simple substances**, or **elements**. Why? Because the elements are those substances that do not result from combination, and do not decompose into other substances. You are to observe examples of the four basic kinds of chemical reactions and take note of the signs or indications that a chemical reaction has taken place, that there has been a change in substance.

Materials

Iron powder
Sulfur
Steel wool (grade #000 or #0000)
Sugar
Hydrogen peroxide
Baker's yeast (as a catalyst)
Copper sulfate
Hydrochloric acid
Magnesium
Lab scale
Iron nail (with 7" string tied to nail head)
4 test tubes (16mm x 150mm)
Test tub rack
Plastic funnel (short stem)
Cotton ball
Magnet (and sandwich bag cover)
Crucible tongs
Aluminum foil (5' x 5' square)
Bunsen burner
Ring stand, ring, and wire gauze
Butane lighter
Thermometer

Method

You are to observe examples of the four basic kinds of chemical reactions and take note of the signs or indications that a chemical reaction has taken place. The four basic kinds of chemical reactions you will observe are:

1. synthesis or combination reactions
2. decomposition reactions
3. single replacement reactions
4. double replacement reactions

Look for signs or indications that a chemical reaction has taken place. Here are some of the most common signs of a chemical reaction:

a) change in temperature – chemical reactions often release or absorb heat. A reaction is called **exothermic** if it produces heat and **endothermic** if it absorbs heat. Combustion is an example of an exothermic reaction. Try to distinguish between the heat required to start a reaction, e.g., lighting something on fire, and the heat produced by the reaction itself, e.g. the heat given off by combustion. A substance that gets so hot that it glows red is often a sign that a reaction gives off heat.
b) change in weight
c) change in color
d) **effervescence** – (production of gas bubbles in a liquid)
e) formation of a **precipitate** (small particles of a solid suspended in a liquid)

Chemical Reactions

The following reactions are presented in the order of complexity, but your lab will begin with a demonstration of the “golden rain” double replacement reaction, and your tutor may ask you to do the single replacement reactions first because the reactions take more time.

Synthesis or Combination Reactions

In a synthesis reaction a compound is made from simpler substances. This kind of reaction is often symbolized by the following formula or equation: $A + B \rightarrow AB$.

Iron + Sulfur → Iron (II) Sulfide

The synthesis reaction between iron and sulfur is a nice way to illustrate the difference between a mixture and a compound since we can chemically combine two of the substances that were mixed together in Part I. Here is the procedure:

1. Take a piece of weigh paper, fold it in half diagonally in both directions to make a simple weigh boat, place it on the lab scale, and press the zero/tare button.
2. Weigh out about 3.5g of iron powder and add it to your mortar.
3. Using the same piece of weigh paper, weigh out about 2g of sulfur and add it to your mortar.
4. Thoroughly mix the two substances using mortar and pestle.
5. Using a short stem plastic funnel, add the mixture to a test tube and plug the end of the test tube with a cotton ball. The purpose of the cotton ball is to absorb any sulfur gas that may be produced as the mixture is heated.
6. Place the test tube in a test tube clamp (tongs) and heat the bottom of the test tube in the hottest part of the flame. Pay careful attention to what happens, especially to the signs of a chemical reaction.
7. The mixture will become red and gummy, and then solid. Continue to heat the test tube until the bottom glows bright red (this is when the reaction takes place).
8. Remove the test tube from the burner and let it cool to room temperature (~five minutes). Do not place the hot test tube on the benchtop. Lay it horizontally across the mortar.

9. Once the test tube is cool, extract the product of the reaction. To do this, roll the test tube up in a paper towel and then strike the wrapped test tube with the pestle to shatter the tube. Carefully unwrap the paper towel and pick out the hard piece of iron sulfide. Discard the broken test tube and any shards of glass in the broken glass waste disposal box.
10. Test the lump of iron sulfide with a magnet. Does it appear to be magnetic?
11. Pulverize the lump of iron sulfide using a mortar and pestle. Now test it with the magnet again. Is it magnetic? If so, is it as magnetic as iron the iron powder? Compare these results with the mixture of iron filings and sulfur in Part I of the lab.

Iron + Oxygen → Iron Oxide

In this reaction you will burn iron. From your experience with iron, this may not seem possible. The way to do it is by using very fine steel wool. Here is the procedure:

1. Weigh a small aluminum weigh boat (or tart tin) and record the weight: ___________ g.
2. Take ¼ of a pad of very fine steel wool and separate the layers of the steel wool from each other to produce a small fluffy ball that will fit inside your weigh boat.
3. Place the ball of steel wool into the weigh boat and weigh it again: ___________ g.
4. Calculate the weight of the steel wool: __________ g. Note the color: __________.
5. Place the weigh boat on top of the wire gauze and light the steel wool with a lighter. Watch what happens.
6. Once the steel wool stops burning, turn over the ball of steel wool being careful not to let any of the fragments fall out of the weigh boat. Now light it again. You may need to light the wool several times to ensure that all of the steel wool has been burned.
7. When all the wool has burned, weigh the weigh boat and wool again: _____________g.
8. Was there an increase in weight? If so, calculate the weight gain: ____________g.
9. Note the color of the burnt wool: ______________. Compare the color of the burnt steel wool with the unburnt steel wool.
10. Are there any other signs of a chemical reaction?

Decomposition Reactions

In a decomposition reaction a compound decomposes into two simpler substances. This kind of reaction is often symbolized by the following formula or equation: AB → A + B.

Sugar → Carbon + Water

In this reaction we will thermally decompose sugar by heating it with a Bunsen burner. Sugar decomposes into carbon and water (there are also small amounts of other substances produced from caramelizing the sugar). Here is the procedure:

1. Cut a piece of 5” square piece of aluminum foil to cover the wire gauze that sits on the ring stand (the lab assistant may have cut the aluminum foil for you in advance).

2. Check that the wire gauze is approximately 1¼" above the top of the Bunsen burner. Adjust the height of the ring if necessary.
3. Weigh a small aluminum weigh boat (or tart tin) and record the weight: __________ g.
4. Add a small amount of sugar (about ½ of a gram) and record it here: _________ g. (Do not add much more than ½ gram sugar, or it will be hard to see the reaction!)
5. Place the weigh boat on top of the foil covering the wire gauze.
6. Take a 400mL beaker and place it upside down on top of the weigh boat.
7. Light the Bunsen burner, place it beneath the wire gauze, and make a note of the time. You should heat the sugar for approximately 1 minute, and no more 1½ minutes.
8. The sugar will melt, caramelize, and eventually turn black. After all of the sugar has turned black, shut off the burner.
9. Note the condensation on the inside of the beaker. This is water. You will also notice an odor that resembles burnt marshmallows (this is a byproduct of the sugar caramelization).
10. Leave the beaker to cool for five minutes before removing it from the burner. **Do not remove the beaker until it has cooled, or you will release some foul smelling gases into the laboratory!**
11. Once the beaker has cooled, remove it from the ring stand and observe the black product in the weigh boat. This is carbon. Let the weigh boat sit for a few minutes for any condensation to evaporate from the carbon (or carefully wipe it off with a paper towel) before weighing it again. Record the weight of the weigh boat and carbon: __________g.
12. Calculate the weight of the carbon: _________g.
13. Calculate the change in weight during the reaction: __________g.

Hydrogen Peroxide → Water + Oxygen

Hydrogen peroxide is a highly reactive substance that is used as a disinfectant, a bleaching agent, and as an oxidizer. It is a relatively unstable substance which, left to itself, slowly decomposes into water and oxygen, especially when exposed to sunlight (this is why it is commonly stored in a brown bottle). In this reaction you will speed up the decomposition of hydrogen peroxide with the help of a catalyst, baker's yeast. (Note: a catalyst is a substance that increases the rate of a chemical reaction without itself undergoing a chemical change).

1. Add a pinch of yeast to a test tube and then fill the test tube ¼ full of hydrogen peroxide.
2. Notice the effervescence. This is oxygen. To test for oxygen, hold a gloved thumb over the mouth of the test tube for a minute or two to collect some of the gas. Then have your lab partner light a wooden splint. Blow out the flame and insert the glowing splint into the mouth of the test tube. If the splint glows brighter, then the gas is oxygen.
3. It can sometimes be hard to observe the change in temperature of a chemical reaction. To test to see whether the decomposition of hydrogen peroxide entails a change in temperature, dump out the test tube in a lab sink, refill the test tube with fresh yeast and hydrogen peroxide, and insert a thermometer into the test tube.

4. Monitor the temperature as the reaction proceeds. Does the reaction produce a change in temperature?

Single Replacement Reactions

Single replacement reactions are those in which a more reactive element replaces a less reactive element in a compound. This reaction is common among metallic compounds when a more reactive metal replaces a less reactive metal (e.g., iron is more reactive than copper). In a replacement reaction, the elemental substance that is being replaced in a compound will suddenly appear as a solid **precipitate**, or through the **effervescence** of a gas. This kind of reaction is symbolized by the following formula or equation: A + BC → B+ AC.

Note: you should do these reactions first so that they can be set aside for later observation.

Iron + Copper Sulfate → Copper + Iron Sulfate

1. Fill a test tube with enough copper sulfate solution to cover the bottom half of the iron nail (about 2 mL). Copper sulfate is poisonous. **Wear gloves and eye protection while handling it!**
2. Polish the nail with a piece of steel wool until it is shiny bright.
3. Using the string attached to the nail head, lower the iron nail into the test tube.
4. Wait 30 seconds and then raise the nail. The pink/brownish coating is copper.
5. Lower the nail again, set the test tube aside, and come back to it later.
6. Periodically check on the nail to observe how much copper has attached to the nail. To estimate how much copper has formed on the nail, gently shake the test tube so that the copper flakes off of the nail.
7. As the soluiton of copper sulfate changes to a solution of iron sulfate, do you see a change in the color of the solution?

Magnesium + Hydrochloric acid → Magnesium Chloride + Hydrogen Gas

1. Fill a test tube about ¼ full of hydrochloric acid. Be careful, hydrochloric acid is a highly corrosive substance. **Be sure to wear gloves and eye protection when handling it!**
2. Drop a one inch strip of magnesium into the test tube and watch what happens.
3. Notice the effervescence.
4. You can test the gas produced for flammability. Hold a gloved thumb over the mouth of the test tube for a minute or two to collect some of the gas. Then have your lab partner light a wooden splint. Remove your thumb and insert the lighted splint into the mouth of the test tube. If the gas is flammable, you should hear a pop sound.
5. Note any other signs of a chemical reaction.

Cleanup

Leave the weigh boat used to burn the steel wool, and the nail with the attached string at your workstation.These items can be reused. Do not discard them.

The iron (II) sulfide, iron oxide, and the weigh boat used to decompose the sugar should be thrown in the trash. Leftover hydrogen peroxide, copper sulfate/iron sulfate can be poured down the drain. Leftover hydrochloric acid/magnesium chloride can be poured down the drain with copious amounts of water.

Demonstration 3: "Golden Rain" Double Replacement Reaction

Double replacement reactions (also known as double decomposition reactions) are those reactions in which the elements contained in two compounds trade places with one another. The most common sign that this kind of reaction takes place is the formation of a precipitate. This kind of reaction is symbolized by the following formula or equation: $AB + CD \rightarrow AD + BC$.

Your tutor will demonstrate the following example of a double replacement reaction.

Lead (II) Nitrate + Potassium Iodide → Lead Iodide + Potassium Nitrate

This is a strikingly colorful reaction of two clear liquids producing a bright yellow precipitate. If the precipitate is heated to near boiling and left to slowly cool it produces amazing crystalline flakes commonly referred to as "golden rain." (Note: Your tutor will likely begin class with this demonstration so that there is time to observe the formation of the "golden rain").

Materials

Lead (II) nitrate
Potassium iodide
400 mL beaker
500 mL Erlenmeyer flask
Warming plate
Water bath
Glass thermometer

1. Before class, the tutor (or lab assistant) will prepare a solution of potassium iodide in a 400 mL beaker by dissolving 0.8g potassium iodide in 200 mL of distilled water.
2. The tutor (or lab assistant) will also prepare a solution of lead (II) nitrate in a 500 mL Erlenmeyer flask by dissolving 0.8 g lead nitrate in 200 mL of distilled water. **Caution: gloves and eye protection should be worn when handling lead (II) nitrate since it is poisonous!** If the lead (II) nitrate solution is cloudy, a drop of acetic acid can be added to clarify the solution.
3. Begin the demonstration by adding a pipette of potassium iodide into the beaker of lead nitrate solution.

4. Swirl the solution. The bright yellow precipitate will be slightly soluble.
5. Add several more pipettes of potassium iodide solution. Watch, and swirl some more.
6. After doing this a few more times, pour the rest of the potassium iodide into the flask.
7. Observe the result, a precipitate of lead iodide.
8. At room temperature, lead iodide is only sparingly soluble, but when heated to near boiling it becomes ten times more soluble. Carefully heat the solution on a warming plate, stirring occasionally with a glass thermometer until the mixture reaches 80 °C. Turn off the warming plate and continue stirring until most of the lead iodide dissolves.
9. Observe the crystallized lead iodide that slowly drops out of the solution as it reaches room temperature. If you are pressed for time, the flask can be placed in a cold water bath to speed up the process.
10. At the end of the class, place the flask of lead iodide under the fume hood.

In the appropriate boxes, note the details of your observations. If you observed a change in temperature, give the details (e.g., “felt warm,” “glowed red,” “increased from 23° to 25°”).

	Temp. change	Weight change	Color change	Effervescence	Precipitate
Reaction of Iron and Sulfur					
Burning Steel Wool					
Decomposition of Sugar					
Decomposition of Hydrogen Peroxide					
Reaction of Iron and Copper Sulfate					
Reaction of Iron and Hydrochloric Acid					
“Golden Rain”					

Report

1. Present your data for Part I in a table so that you may easily see the results of your procedures. You may use the table printed in Part I.
2. In those cases where you successfully separated a mixture, be sure to note which physical properties you relied upon to make the separation. (This information does not necessarily have to be included in the table. It may be presented separately.)
3. Present the results of your observations of the various chemical reactions in Part II in a data table, paying special attention to the signs of a chemical reaction. You may use the table on the previous page.

Questions and Problems

1. There seem to be important differences between the way salt dissolves in water and the way a metal dissolves in an acid? How would you describe the difference?
2. What evidence is there to distinguish the mixture of iron and sulfur in Part I and the **product** of heating iron and sulfur together in Part II?
3. Consider this position: "When A is dissolved in B, some claim A is still present as such, though to all appearances A has disappeared. For example, table salt (A) dissolved in water (B) gives a solution of A in B even though some characteristics of salt (e.g. its solidity at room temperature) have disappeared. I maintain that 'dissolving' A in B destroys both A and B as such, and a new substance, C, is formed. Under certain conditions C may in turn be destroyed and A and B produced out of it; e.g., the substance C on boiling is converted into A (salt) and eventually B (steam, which may be changed into water)." Do you agree with this position, viz., that dissolving A in B destroys both A and B and that a new substance, C, is formed? If not, how might you defend the actual presence of A throughout these changes?
4. Do you have any reason to believe that the substances you observed in Part II are elements? Why, or why not? When should you assume that a given substance is an element rather than a compound? Is it more scientific to assume a substance is elementary until proven otherwise, or a compound until proven elementary?

Experiment 2: Weight Change of a Burning Metal

One of the focal points of the dispute between Lavoisier, Boyle, and the phlogiston theorists was how to explain the weight change of a burning metal (or what the early chemists called “calcination”). In this experiment we will observe this change. Since the precise amount of weight gained by a burning metal played an important role in the dispute, we will attempt to measure accurately the weight gained.

Materials

2 strips of magnesium 6” long
Crucible
Crucible tongs
Ring stand with ring
Bunsen burner
Lighter (or striker)
Clay triangle
Steel wool
Dry paper towel
Scoopula
Lab scale (one that measures to .01g)

Safety Notice

Avoid inhaling any smoke produced by the burning magnesium. Always use crucible tongs when handling the hot crucible or lid.

Method

In this experiment we will be burning strips of magnesium metal in a porcelain crucible, a small vessel that can withstand very high temperatures. When magnesium burns with a ready supply of air it burns with an intense bright light (which can damage the eyes) and gives off white smoke. To measure accurately the weight gained by the burning magnesium, it will be necessary to minimize the amount of smoke given off during the calcination. We will do this by allowing a small crack between the crucible and its lid, which will effectively control the amount of air in the crucible. Here is the procedure:

1. Make sure the crucible is clean and empty. Do this by gently cleaning it with a dry piece of steel wool (do not use water) and wiping it out with a dry paper towel.
2. Place the empty crucible in the clay triangle on the ring stand and adjust the height of the ring and clay triangle such that the bottom of the crucible is about 1” above the top of the Bunsen burner.
3. Using a lighter (or striker), light the Bunsen burner and adjust the flame so that there is a well-defined inner blue cone approximately 1” high.
4. Place the Bunsen burner directly beneath the crucible and heat it (without the lid) for two minutes to burn off any residual moisture or other impurities.
5. After two minutes, using crucible tongs place the lid on the crucible and proceed to heat for another two minutes.

6. Turn off your Bunsen burner and let the crucible cool to room temperature. This should take about ten minutes.
7. Once it has cooled, weigh the crucible. **It is important that the crucible be cool before it is weighed, as a hot crucible will weigh less than a cool one!** Record the weight of the crucible and lid here: _____________ g.
8. Polish one of the magnesium strips with steel wool until it is shiny bright.
9. Shape the strip of magnesium ribbon into a coil or spiral that will fit into the bottom of your crucible. (To shape the magnesium ribbon into a coil, try wrapping it around a pencil.)
10. Add the magnesium to the crucible and weigh the crucible, lid, and magnesium. Record the weight here: ______________ g.
11. Calculate the weight of the magnesium strip added to the crucible: _______________ g.
12. Place the crucible in the clay triangle and adjust the lid so that it is slightly askew, leaving a small gap for air to enter into the crucible. **It is important to leave only a small gap between the lid and the crucible in order to control the amount of air that enters the crucible**. If the gap is too large, the magnesium will burn more intensely and smoke will escape from the crucible, which will mean less accurate results. The lid should look something like this:

13. Light the Bunsen burner and begin heating the crucible. After heating the crucible for approximately one minute, the magnesium should begin to burn and you should be able to see a red glow coming from the gap between the lid and crucible.
14. Continue to heat the crucible for a full five minutes.
15. After five minutes, check whether the magnesium ribbon has finished calcinating. To do this, lift the crucible lid with the crucible tongs and check to see whether the magnesium is still glowing red (note: the crucible itself may be glowing red from the heat of the burner, but once the ribbon is fully calcinated it will no longer glow red). If the magnesium is still glowing red, place the lid back on the crucible and check it again in one minute.
16. Once the magnesium seems to be fully calcinated, place the lid back on the crucible and heat it for an additional two minutes.
17. Turn off the Bunsen burner and let the crucible cool to room temperature before weighing it. **Again, it is important that the crucible is cool before weighing!** Record the weight of the crucible and magnesium calx here: _______________ g.

18. Calculate the weight gain: ______________ g.
19. Determine by what percentage the metal increased in weight (weight gained ÷ weight of the magnesium x 100): ____________ %.
20. Record your results on the blackboard and in the table on the following page. Make a note of the results of your fellow students.
21. Using the scoopula, **gently** scrape out the burnt magnesium from the crucible and dispose of it in the waste container. **Do not scrape out the crucible aggressively in an attempt to remove the stain in the bottom of the crucible!** It is normal for the crucibles to become stained by burning magnesium.
22. Gently clean out the crucible with steel wool, and wipe it out with a dry paper towel.
23. Repeat steps 7-21 with the second strip of magnesium.
24. When you are finished, clean out your crucible and wipe down the workstation with a paper towel.

Team	Crucible + Lid	Crucible + Lid + Magnesium	Magnesium	Crucible + Lid + Calx	Weight Gain	% Increase
1.						
2.						
3.						
4.						
5.						
6.						
7.						
8.						
Class Average	▨	▨		▨		

Demonstration 4: Calcinating a Metal in a Sealed Vessel?

With time permitting, your tutor (or a lab assistant) may calcinate a metal in a *sealed* vessel in a demonstration similar to Lavoisier's calcination of tin in a sealed vessel. From our gas laws, we know that any gas trapped in the heated vessel will expand with the heat increase and the pressure will also therefore increase, so attempting to burn a metal in a sealed container can be dangerous and Lavoisier tells us as much. To minimize the risk of explosion, this demonstration will be performed using steel wool and a magnifying lens rather than using magnesium and a Bunsen burner. Since steel wool requires less heat to ignite, we can burn it easily using a powerful magnifying glass (or what Lavoisier refers to as a "burning glass"). In this case, we will be using a Fresnel lens, a powerful but relatively inexpensive magnifying lens.

Materials
Steel wool
1 liter-sized Mason jar (or any large wide-mouthed jar that seals well)
Fresnel lens (8 x 10)
Lab scale (one that measures to .01g)

Here is the procedure:

1. Weigh an empty mason jar on a lab scale and record the weight: _____________ g.
2. Take about ¼ of a pad of fine steel wool (#0000) and carefully separate the filaments so that it can be shaped into a fluffy ball.
3. Place the steel wool into the jar, tighten the lid, and then weigh it again: _____________ g.
4. Calculate the weight of the steel wool: ______________ g.
5. Find a sunny spot outside and use the Fresnel lens to focus the light of the sun onto the steel wool. It should quickly catch fire and spread through the ball. You might need to try several times to ignite as much of the steel wool as you can.
6. Allow the jar to cool to room temperature and then weigh it again: ______________ g. Does it weigh the same? Now unseal the jar and record the weight: _____________ g.

Report

1. Present your data and that of your classmates in a table so that you may easily see the results of all of your procedures.
2. Be sure to include the calculation of the weight increase percentages.
3. Compare the percentage weight gains of your two trials. Are they consistent? Are your results consistent with the other lab teams? What are the likely sources of error in your data, or those of your classmates?
4. If the percentage weight gain was relatively consistent, does this suggest anything regarding the composition of the magnesium calx.
5. What were the results of the demonstration? Did the sealed vessel weigh the same before and after the calcination? Did the sealed vessel weigh the same once it was opened? Do the results fit with Lavoisier's experiment with calcinating tin in a closed vessel?

Questions and Problems

1. How would a phlogistonist explain what happened to the burning metal? How would Boyle explain it? How would Lavoisier explain it? Do these different explanations equally fit the results of this lab?
2. What is the significance of the results of the demonstration?

Experiment 3: Copper Oxidation and Reduction

In previous labs we observed what the early chemists called "calcination," which is the production of a metallic oxide (or "calx") through heating or burning a metal in the presence of "vital air" (which Lavoisier calls "oxygen"). We will begin the present lab with the calcination of a metal, and then we will reduce the metal oxide back to pure metal by heating it in the presence of carbon, a method of reduction frequently employed by the early chemists. This reduction yields not only a pure metal but also a gas that the early chemists called "fixed air," or what Lavoisier will call "carbonic acid."

Synopsis

In this lab we will work with copper metal, observing its transformations from pure metal to copper oxide and then back to pure copper again. The lab has two parts. In Part I we will oxidize copper powder by heating it in a crucible to produce black copper oxide. In Part II we will reduce the copper oxide to pure copper by heating it with carbon powder. To verify that the gas produced by the reduction is carbonic acid we will pass the evolved gas through lime water.

Materials

Copper powder (fine mesh)
Carbon powder[1]
Lime water (filtered solution of calcium hydroxide)
Crucible
Crucible tongs
Ceramic tile (or porcelain tile)
Ring stand with ring
Bunsen burner
Lighter (or striker)
Mortar and pestle
Clay triangle
Lab scale (one that measures to .01g)
Scoopula
Plastic funnel (short stem)
2 test tubes (16mm x 150mm)
Small utility clamp (suitable for clamping test tube)
Rubber stopper w/bent glass delivery tube
Safety glasses
Dry paper towel

Safety Notice

Copper powder and black copper oxide are poisonous if inhaled, ingested, or absorbed through skin. Avoid dispersing the dust and wash your hands if you get it on your skin.

---

[1] Carbon powder is hygroscopic (i.e., it tends to absorb water) and should be dried thoroughly before this experiment to achieve the best results since the water absorbed by the carbon will distort the weight of the carbon and add moisture to the final product. Carbon powder should be placed in a drying oven at 150°C for 6-8 hours. After drying, it can be stored at room temperature in a desiccator with a drying agent.

Part I – Production of Copper Oxide by Calcination

When heated in the presence of oxygen, copper metal reacts to form copper oxide. This process is what Lavoisier and the other early chemists called calcination. The word equation for the reaction is Copper + Oxygen → Copper Oxide. Because only the surface of copper metal will react to form copper oxide, you will use a finely powdered copper in order to maximize its surface area. Here is the procedure:

1. First, weigh an empty crucible (without the lid) and record the weight here: _____________ g.

2. Using a clean scoopula, add approximately two grams of copper powder to your crucible and weigh again: ______________ g.

3. Calculate the exact weight of the copper added to your crucible: _______________ g.

4. Place the crucible in the clay triangle and adjust the clay triangle such that the bottom of the crucible is approximately 1” above the top of the Bunsen burner. Then tilt the crucible toward you so that you can observe the copper powder as it is being heated in your crucible.

5. Using a lighter (or striker), light the Bunsen burner and adjust the flame so that there is a well-defined inner blue cone approximately 1” high.

6. Place the Bunsen burner beneath the crucible and begin heating the copper powder.

7. Watch carefully for the surface of the copper powder to begin oxidizing (it will turn dark gray). Once the surface of the copper powder has oxidized, immediately turn off the burner.

8. Wait two minutes before handling the red hot crucible. Then, using the crucible tongs, transfer the crucible to the ceramic tile on your benchtop. While holding the crucible with your crucible tongs, carefully stir the copper powder with the blunt end of your scoopula, breaking up any larger chunks with the convex side of the scoopula. If you are having difficulty breaking it up, you may pulverize it with a mortar and pestle.

9. Once the copper powder has been thoroughly mixed, transfer the crucible back to the clay triangle, light your Bunser burner, and begin reheating the copper powder. Note: try to avoid heating the crucible so intensely that it glows red-hot.

10. After heating the copper powder for several minutes, turn off the Bunsen burner and again stir the copper powder as detailed in step 8. You will heat the copper powder a total of three or four times to ensure that the copper powder has been fully oxidized.

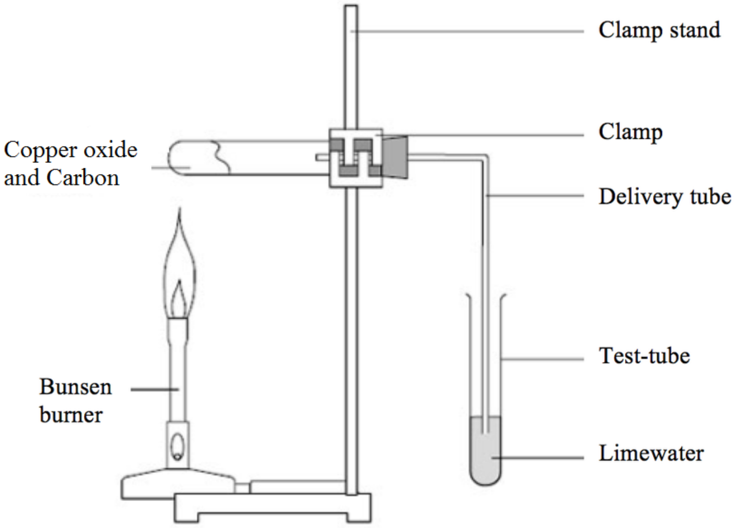
11. After your final heating, turn off the Bunsen burner and let the crucible cool to room temperature (this will take approximately ten minutes). Once it is cool, remove the crucible from the clay triangle and weigh the crucible again: _____________ g.

12. Calculate the weight of the copper oxide: _____________ g.

Part II – Reducing the Copper Oxide by Heating with Carbon

In Part II copper oxide is reduced to copper metal by heating it with carbon. This produces a gas that Lavoisier calls "carbonic acid gas" (what we now call "carbon dioxide"). As Lavoisier notes, when this gas is passed through filtered lime water it produces a milky white precipitate. This precipitate is limestone (calcium carbonate). Here is the procedure:

13. Calculate how much carbon powder to mix with the oxide. The ratio of copper oxide to carbon should be 13:1. Amount of carbon to add to mixture: _____________ g.
14. Using the lab scale, weigh the appropriate amount of carbon and add it to your crucible containing copper oxide.
15. Using the scoopula, carefully mix the copper oxide and carbon in the crucible.
16. Pour the mixture into a test tube using a plastic funnel. If any of the mixture sticks to the sides of the crucible, scrape it off using the scoopula.
17. Loosen the clamp attaching the ring to the ring stand and rotate it out of the way. Then, using a small utility clamp, loosely clamp the test tube containing copper oxide and carbon to the clamp stand so that it is parallel to the benchtop. The clamp should grip the test tube near the mouth of the tube, otherwise the heat from the burner will damage the clamp. Adjust the height of the clamp so that the end of the test tube is approximately 1½" above the top of the Bunsen burner. Insert the rubber stopper into the test tube with the glass delivery tube facing down. Fill a second test tube about 2/3 full with lime water and insert the end of the delivery tube into this test tube. Make sure that the test tube with the copper oxide can rotate easily so that the delivery tube can be lifted out of the test tube containing the lime water once the reaction is complete. Your setup should look something like this:



To avoid 'suck-back' the delivery tube must be lifted from the limewater as soon as heating ends.

18. Light the Bunsen burner and begin heating the mixture of copper oxide and carbon in the hottest part of the flame for several minutes.
19. As the mixture is heated and the reaction begins to take place you will see the gas produced bubbling through the lime water. Observe the effects of the gas on the lime water.

20. The reaction is complete when the gas no longer bubbles through the lime water.
21. Rotate the delivery tube up and remove the test tube holding the lime water, then turn the Bunsen burner off. **Remove the test tube containing lime water first, or the water will be sucked back into the test tube containing the copper and cause the test tube to crack**.
22. While the test tube is cooling, clean your crucible with a dry paper towel.
23. Once the test tube has cooled to room temperature, remove the test tube from the clamp and tip out the contents into the crucible. Carefully break up any lumps with the blunt end of the scoopula. You should be left with a pinkish brown substance. This is copper!
24. Weigh the crucible again: _____________ g.
25. Calculate the weight of the copper: _____________ g.
26. Calculate any change in the weight of the copper from start to finish: _____________ g.
27. Calculate the percentage increase/decrease (weight change ÷ weight of copper x 100): ____________ %.
28. Record your results on the blackboard and in the table below. Make a note of the corresponding results of your fellow students.

Team	**Copper (Start)**	**Copper Oxide**	**Carbon**	**Copper (Finish)**	**Weight Change**	**% Increase/ Decrease**
1.						
2.						
3.						
4.						
5.						
6.						
7.						
8.						
Class Average	▨	▨	▨	▨		

Cleanup

Clean your work station before you leave: Dispose of your copper in the beaker labeled "waste copper" (not down the drain). Wipe out the crucible and mortar with a dry paper towel. Rinse out your test tubes with water. Wipe your bench top with a damp paper towel.

Questions

1. During the oxidation of the copper in Part I of this lab, you may have observed that the copper does not immediately change from pink to black, but appears to change first into an intermediate colors. Why might this be?
2. How would Lavoisier describe the change in the carbon in Part II of this lab?
3. Which of the four basic kinds of chemical reactions we observed in Experiment 1 can be applied to the copper reduction in Part II of this lab?
4. How close was the weight of the copper yielded by the reduction to the weight of the copper with which you started? If the two measurements differ significantly, can you think of any reasons why they differ? Did the percent increase, or decrease, differ significantly from that of the class average? If so, why might that be?

Brownian Motion Demonstration (optional)

Synopsis

Brownian motion is the name for the random movement of minute particles suspended in a liquid as a result of continuous bombardment from particles in the surrounding medium. It is named from Robert Brown, a 19th century botanist who, while observing pollen grains in water under a microscope, noticed the random jittery movement of particles released from the pollen grains. Although Brown did not offer a theory to explain the random motion, Albert Einstein wrote a paper in 1905 explaining in detail how Brownian motion can be explained by the molecular theory of heat—the theory that heat is the motion of particles. Einstein predicted that the random motions of molecules in a liquid (e.g., water) would cause the random movement of larger particles suspended in the liquid which could be observed under a microscope.

Method 1

Although Brownian motion is best observed under a microscope, there are many of examples of Brownian motion familiar to us from common experience, e.g., the dispersion of smoke in a room, the rapid diffusion of perfume and other odors, and the random glittering motion of dust motes in the air noted by Lucretius in *On the Nature of Things*, Bk. II, 115-142. Hence, we will begin by observing an example of Brownian motion close to common experience: the motion of small particles of food coloring suspended in very hot water.

Materials

2 large 400 mL beakers
Distilled water
Bunsen burner
Lighter (striker)
Ring stand, ring, and wire gauze
Thermometer

Procedure:

1. Fill two 400 mL beakers with distilled water.
2. Heat one of the beakers of water over a Bunsen burner until it reaches approximately 80 °C.
3. Let the two beakers sit undisturbed for 3-5 minutes to ensure that there are no obvious movements in the water.
4. Add one drop of food coloring to the center of each beaker. Be sure to drop the food coloring from the same height for each beaker.
5. Observe what happens. Since the particles of food coloring are more dense than the water they tend to sink to the bottom of the beaker, but you should notice that while the particles of food coloring in the cold water slowly sinks to the bottom, the food coloring in the very hot water is rapidly dispersed with a random motion.
6. After the food coloring has completely dispersed in the hot water, add an additional drop of coloring to the two beakers.

Question

1. Does this demonstration provide convincing evidence that heat is a motion of particles? Can the phenomena be explained in some other way, e.g., thermal convection currents?

Method 2

Brownian motion is best observed under a microscope. In this case we will observe the tiny fat particles suspended in homogenized milk under a compound microscope. A compound microscope has two kinds of lenses, a 10X lens in the eyepiece (called an ocular lens) and a set of objective lenses that point directly at the stage where the slides are placed. By using both lenses in tandem, the microscope can achieve hight levels of magnification. A 10X ocular lens with a 40X objective lens yields a magnification of 400X!

Materials

Compound microscope
Clean slide and coverslip
Homogenized milk
Distilled water
Glass stir rod
Paper towel

Procedure:

1. Put a very tiny amount (less than a drop) of milk onto a clean slide.
2. Dilute the milk by adding a drop of distilled water (diluting the milk makes the fat particles easier to see). Mix the milk into the water.
3. Place a coverlip over the mixture of milk and water gently press it down onto the mixture. Remove any excess liquid with a dry paper towel.
4. Put the slide under the microsope, turn on the microscope, and adjust the light to maxium brightness.
5. Begin by focusing the fat droplets using the 10X objective lens (the lens with yellow lettering). Under the 10X objective lens, the fat particles appear as tiny specks that are barely visible.
6. Move to the 40X objective lens (the lens with blue lettering). You should see tiny droplets of milk fat moving in random directions. (At first, the tiny spheres may appear to be moving in one direction. This is not Brownian motion, but currents in the liquid. This should disappear quickly.)

Questions

1. According to the explanation of Brownian motion under the microscope, the fat globules move with a random motion because they are being bombarded by water molecules that are much smaller, and moving much faster, than the particles of milk fat. Is this explanation convincing?
2. Does the demonstration of Brownian motion under the microscope (as opposed to the demonstration using food coloring) provide more convincing evidence for the molecular theory of heat?

Demonstration 5: Composition of Water from Hydrogen and Oxygen Gas

Toward the end of your discussion class on chapter 8, a laboratory assistant will present to you a modified version of Lavoisier’s “experiment 4.” Using an electric spark-generator, your tutor will demonstrate that when hydrogen and oxygen gases are combined in the ratio of 2 to 1, by volume, water can be produced by the rapid oxidation of the hydrogen effected by the spark. (This experiment may also be postponed until after Gay Lussac has been read and the decomposition of water is presented.)

Experiment 4: Some Common Types of Pure Substances

To gain familiarity with substances and processes mentioned in the *Elements of Chemistry,* and to prepare for reactions to be mentioned in subsequent readings, you will observe several demonstrations and carry out several experiments that will illustrate the main types of pure substance: metals, non-metals, acids, bases, and salts.

- Metals have been shown not to decompose, and thus, by Lavoisier's standard, are elements.
- Non-metals fall into Morveau's first and second classes of substances (excepting light and caloric), and have also been proven to be elements. Non-metals are not lustrous, are rarely gray, are not malleable, and are usually poor conductors of heat.
- Acids are composites with distinctive characteristics. (One task in this experiment is to determine some characteristic property or properties of acids. You should also note how acids are formed.)
- Bases are composites with distinctive characteristics. (One task in this experiment is to determine some characteristic properties of bases. You should also note how bases are formed.)

  Note: To avoid confusion, we should note that the word “base” is no longer used in the broad sense that Lavoisier uses. It is now confined to what he calls a “salifiable base.” He generally uses the term “base” in a broader sense, but always in the sense of something that combines with something else. Thus oxygen is the “base” of oxygen gas, the other constituent being caloric. You will notice later that Berthollet uses “base” in the newer, narrower sense, which is also the sense in which the term is used in this experiment.
- Salts are composites usually produced by the reaction of an acid and a base, but that may also be produced by the reaction of an acid and a metal. When not dissolved they are crystalline solids.

Safety Notice

The acids used in this laboratory are corrosive, and the bases are caustic. Unless otherwise directed, wear gloves and safety glasses at all times.

Part I

You will observe some of the sensible properties of acids and bases, the neutralization reaction between an acid and a base, and the reaction of certain metals with both water and acid.

Demonstration: Sensible properties of Acids and Bases (tutor demonstration table)

Synopsis

As an introduction to acids and bases, your tutor will discuss some of the sensible properties of acids and bases, and demonstrate how to test whether a substance is an acid or a base using litmus paper.

Materials

Caustic soda (sodium hydroxide, 1M)
Potash (potassium hydroxide, 1M)
Volatile alkali (ammonia, 1M)
Muriatic acid (hydrochloric acid, 1M)
Sulfuric acid (1M)
Nitric acid (1M)
Acetic acid (1M)
1 Liter Seltzer water
7 test tubes (16mm x 150mm)
Test tube rack (plastic rack with spaces for 8 test tubes)
Red and blue litmus paper
3mL disposable pipette
250 mL beaker (for rinsing pipette)
50 mL beaker (for diluted acetic acid)

Before class, the tutor (or lab assistant) will fill seven test tubes with approximately 5 mL of the following: muriatic acid, sulfuric acid, nitric acid, acetic acid, caustic soda, potash, and volatile alkali. (This should be prepared by the lab assistant before class begins.)

Solubility

All acids are readily soluble in water, and the four acids in the test tubes in front of you (viz., muriatic acid, sulfuric acid, nitric acid, and acetic acid) are dissolved in water. Some bases are readily soluble in water including the three bases in the test tubes in front of you (viz., caustic soda, potash, and volatile alkali). They are also dissolved in water. Unlike acids, there are some bases that are only sparingly soluble in water (e.g., calcium hydroxide, magnesium hydroxide). You will have a chance to observe bases that are only sparingly soluble in water later in this lab.

Color

Note that the acids and bases are colorless, which is typical of most acids and bases when dissolved in water. Concentrated acids sometimes turn yellow (or reddish), but this is due to impurities. In their solid form, bases are typically white.

Odor

Acids and bases do not, as a general rule, have a characterstic odor. As Lavoisier points out, however, both muriatic acid (hydrochloric acid) and volatile alkali (ammonia) are highly volatile and have strong penetrating odors. Pass around the test tubes of muriatic acid and volatile alkali so that everyone can get a chance to smell these two substances. Waft the vapor from the liquid toward your nose, being careful not to place your nose too close to the mouth of the test tube.

Taste

Acids have a sour or tart taste, and many foods have a sour or tart taste due to the presence of an acid. The sour taste of vinegar is due to “acetic acid,” the sourness of lemons, limes, and grapefruit is due to “citric acid,” the tartness of green apples is due to “tartaric acid,” the sourness of sour cream and sour milk is due to “lactic acid,” and the slightly sour taste of Seltzer water is due to “carbonic acid gas.” The sour taste of acids is probably the most characteristic sensible property of acids. Indeed, the German word for acid is “sauer” (pronounced just like the English word “sour”). Taste a sample of diluted acetic acid. Wet the tip of your finger in the diluted acid and place a small amount on your tongue. Rinse your palate with water when you are done. Taste a sample of Seltzer water.

Bases are bitter tasting, although there are very few foods that are alkaline. The bitterness of some raw vegatables such as wild salad greens and artichoke is due their alkaline properties. Baking soda (sodium bicarbonate) is a weak base. Antacids are often made from milk of magnesia (magnesium hydroxide), which is a base. The three bases in the test tubes (i.e., caustic soda, potash, and volatile alkali) are all strong bases and caustic. **Do not taste any of the bases!**

Touch

Acids do not have an especially noteworthy feel, but bases feel slippery and soapy. This is not accidental: soaps and cleaning agents are made from bases. Soap is made from caustic soda; liquid soaps are typically manufactured from potash; and volatile alkali (ammonia) is used as a cleaning agent. Bleach is another common househould cleaner that is a strong base and has a characteristic slippery or soapy feel. Feel the sodium hydroxide. Place your thumb over the test-tube and invert it. Rub your fingers together and experience the soapy feel characteristic of bases. Wash your hands with water when you are finished.

Testing with litmus papers

The idea of using litmus paper for testing whether a substance was an acid or a base was introduced by Robert Boyle. Litmus paper is ordinary paper treated with an organic dye (sometimes from the pigments of red cabbage or violet petals) which reacts distinctively with acids and bases. Red litmus paper turns blue in the presence of a base (or alkali) and remains unchanged in the presence of an acid. Blue litmus paper turns red in the presence of an acid and remains unchanged in the presence of a base (alkali). Your lab tutor will demonstrate how to test for acidity or alkalinity using litmus papers.

Here is the procedure:

Fill a 250 mL beaker with distilled water. This will be used to rinse the disposable pipette when testing the acids and bases with litmus paper. Test a sample of Seltzer water and each of the seven liquids in the test tubes with red and blue litmus papers by taking the end of a clean pipette, dipping it in the liquid, and touching the end of the pipette to the litmus paper. Rinse the pipette with distilled water before testing the next liquid. For comparison’s sake, you should also test distilled water with red and blue litmus papers.

Reactions involving Acids and Bases

Materials

Caustic soda (sodium hydroxide, 1M)
Muriatic acid (hydrochloric acid, 1M)
Acetic acid
Distilled Water
Calcium metal
Magnesium strip 1” long
6 test tubes (16mm x 150mm)
Test tube rack (wooden rack)
Red and blue litmus paper[1]
50 mL beaker
2 disposable pipettes (3mL)
250 mL beaker with distilled water (for rinsing pipette)
Bunsen burner
Ring and wire gauze
Extra test tube (for collecting gas)
Lighters
Wooden splints

Section 1: Neutralization Reaction of an Acid and a Base

Synopsis

You will observe a neutralization reaction in which a strong acid and a strong base react to form water and a neutral salt. In this case, you will observe the reaction of caustic soda and muriatic acid which together produce table salt (muriate of soda) and water. The word equation for the reaction is:

$$\text{Caustic Soda} + \text{Muriatic Acid} \rightarrow \text{Muriate of Soda} + \text{Water}.$$

Here is the procedure:

1. Add 5mL of caustic soda to a test tube and 5mL of muriatic acid to another test tube.
2. Using a clean disposable pipette, add 3 mL of caustic soda to a 50 mL beaker.
3. Using a clean disposable pipette, add 2mL of muriatic acid to the beaker. At this point the mixture should still be basic. Verify this by testing with litmus paper.
4. Now add muriate acid a few drops at a time. Test with blue litmus paper after each addition of drops by taking the end of a clean pipette, dipping it in the mixture, and touching the end of the pipette on the litmus paper (allowing you to use one piece of litmus to test the solution several times). Rinse the pipette with distilled water before testing again. Continue to do this until the blue litmus paper indicates acidity.
5. At this point, the liquid in the beaker is slightly acidic. Once you reach this point, add caustic soda one drop at a time until the blue litmus paper indicates no change in the color of the paper.

---

[1] Litmus paper is ordinary paper treated with an organic dye (sometimes from the pigments of red cabbage or violet petals, discovered by Boyle to react distinctively with acids and bases). “Litmos” (Old Norse): “color moss.”

This is the neutralization point.

6. Using a Bunsen burner, evaporate the liquid and examine the residue. This is table salt.
7. *If* you are confident that you reached a neutralization point when producing the Muriate of Soda, you *may* taste a *tiny* amount of it to see if it indeed seems to be table salt.

Section 2: The Reaction of a Metal and Water

Synopsis

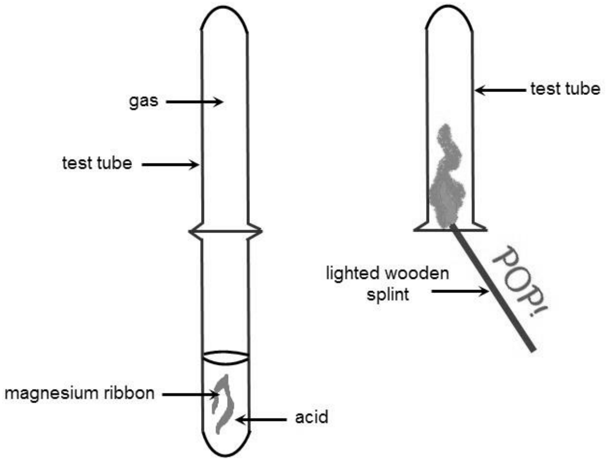
Some reactive metals react with water to produce a metal hydroxide (which is a base) and hydrogen gas. In this case, you will observe the reaction of calcium and magnesium with water. The word equations for these reaction are:

Calcium + Water → Calcium Hydroxide + Hydrogen.

Magnesium + Water → Magnesium Hydroxide + Hydrogen.

Here is the procedure:

1. Fill two test tubes with approximately 5 mL of distilled water and place them in the test tube rack.
2. Place a small piece of calcium into the test tube and a 1” strip of magnesium to the other. **Note: do not hold the test tubes in your bare hands, or you may receive a severe burn!**
3. Note any signs of a chemical reaction (e.g., the evolution of a gas, a change in temperature, the appearance of a precipitate, a change in color).
4. If a gas evolves, test it for flammability. Collect some of the gas by holding an inverted test tube over the mouth of the test tube producing the gas, then have your lab partner insert a lighted wooden splint into the mouth of the inverted test tube. See diagram below. A flammable gas produces a squeaky pop sound.



5. Test the resulting solution with red and blue litmus papers.
6. Record your observations in the table on p. 45.

Section 3: The Reaction of Metals and Acids

Synopsis

Although relatively few metals react violently with water, many metals react with acid to produce a salt and hydrogen gas. You will observe two metals, magnesium and calcium, each reacting with two different acids muriatic acid (hydrochloric acid) and acetic acid. In the process you will observe that some metals react more violently than others. (Indeed, metals form a reactivity series, from metals that are the most reactive to those that are the least reactive.) You may also observe that one acid is stronger than another, i.e., it causes a more violent reaction than another.

1. Fill two tubes with approximately 5 mL of muratic acid and place them in the test tube rack.
2. Add one small piece of calcium to one test tube and a 1” strip of magnesium to the other.
3. Note any signs of a chemical reaction (e.g., the evolution of a gas, a change in temperature, the appearance of a precipitate, a change in color).
4. If a gas evolves, test it for flammability.
5. Fill two tubes with approximately 5 mL of acetic acid and repeat steps 2-4.
6. Note if one metal reacted more violently than the other, or if one acid is stronger than the other.
7. How does the reaction of magnesium and calcium with water compare to its reaction with the two acids?
8. Record your observations in the table on the following page.

		Effervescence	Temp. change	Precipitate	Litmus Paper	Reactivity
Distilled Water	Magnesium					
	Calcium					
Muriatic Acid	Magnesium					
	Calcium					
Acetic Acid	Magnesium					
	Calcium					

Experiment 4: Survey of Some Common Substances (continued)

Part II

You will examine the colors of the flames of several salts when heated to incandescence, and observe several replacement reactions. Time permitting, you will also observe demonstrations in which you will see the reaction first of a metal, and then of a non-metal, with pure oxygen.

Safety Notice

In Part II of this lab you will be working with several toxic salts. Be sure to wear gloves and safety glasses. Barium salts (barytes) are especially toxic and have their own waste container. Be sure to notify the lab instructor if the barium salts are spilled.

Demonstration: Flame Testing of Various Salts (tutor demonstration table)

Synopsis

In this section you will observe a method called "flame testing," which shows the distinctive colors of various salts when they are heated to incandescence in a flame. Flame testing involves a loop of platinum or nichrome wire mounted on a handle: this is called an inoculation loop. Platinum and nichrome wire are corrosion resistant and have high melting points, so you can be sure that the color that you see is the color of the salt and not of the wire. Here is the procedure:

Materials

Sample salt solutions in small vials (listed in step 6)
Innoculation loop
100 mL beaker ½ full of concentrated muriatic acid
Meker burner (burner with a wider top with a grid producing an array of many small flames)
Lighter

Here is the procedure:

1. Dim the lights to make the colored flames more evident.
2. Place the metal part of the inoculation loop into the flame of the Meker burner about an inch above the grid of the burner. If there is a significant change in the color of the flame,[2] purify the loop by heating it to incandescence, and then dipping it in a solution of concentrated muriatic acid. (**Caution: concentrated Muriatic Acid can cause severe chemical burns, so place the beaker of concentrated acid in a place where it will not be easily spilled**.)
3. When dipping the flame loop into the beaker of acid, be sure to insert only the metal part of the inoculation loop into the acid. If the glass portion of the loop becomes hot, it may crack if it is dipped into the beaker of room temperature acid.
4. After dipping the loop in muriatic acid, heat it to incandescence again. Repeat this process until there is no significant change in the color of the flame.
5. Dip the loop into the salt solution to be flame-tested and insert the inoculation loop into the

---

2. The orange-yellow flame which sometimes accompanies the incandescent wire is due to the incandescence of carbon particles produced by the decomposition of the burner gas (a carbon-hydrogen compound) when it comes in contact with the very hot wire. As often this flame will not go away of itself, you might avoid it by finding a part of the burner's flame in which it does not appear, and performing your flame tests by putting the loop in that part of the flame.

burner flame.

6. Note the brief change in the color of the flame, and also note the intensity of the color.
7. The following substances will be flame tested. Note the flame color in the table below:

Salts	Flame Color
Muriate of Potash (Potassium Chloride)	
Nitrate of Potash (Potassium Nitrate)	
Muriate of Soda (Sodium Chloride)	
Nitrate of Soda (Sodium Nitrate)	
Sulfate of Copper (Copper Sulfate)	
Muriate of Calcium (Calcium Chloride)	
Lithium Carbonate	
Muriate of Barytes (Barium Chloride)	

Section 4: Precipitate Reactions

Synopsis

You will observe several precipitate reactions. A precipitate reaction is a kind of replacement reaction in which two water soluble salts (or a water soluble salt and an acid) are mixed with one another and a solid forms from the reaction between the two. The solid is called the **precipitate** and the remaining fluid is called the **supernatant liquid.** These reactions are called replacement reactions because the elements contained in the two salts trade places with one another. As we will see in future readings, replacement reactions play a significant role in the early modern chemists' formation of tables of equivalency. When a given quantity of one substance is replaced by a given quantity of another substance, the amounts of those two substances, even if not equal, may be said to be "equivalent."

Why do the elements in one substance trade places with the elements in another substance? Chemists explain replacement reactions by saying that an element in one substance has a greater "affinity" for an element in another substance thus causing them to react in such a way that the element in one substance trade places with the element in another.

Materials[3]
Nitrate of potash (potassium nitrate)
Sulfate of potash (potassium sulfate)
Sulfate of copper (copper sulfate)
Sulfuric acid
Muriate of barytes (barium chloride) – in a dropper bottle
Nitrate of barytes (barium nitrate) – in a dropper bottle
4 test tubes (16mm x 150mm)
Test tube rack
Funnel
Filter paper
Wash bottle with distilled water

Here is the procedure:

1. Fill four test tubes with approximately 2 mL of nitrate of potash, sulfate of potash, sulfate of copper, and sulfuric acid.
2. Add a few drops of muriate of barytes to each of the four original test tubes.
3. If a precipitate forms, note the color. Keep in mind that a solid precipitate may be of microscopic size, and so may appear merely as cloudiness in an otherwise transparent liquid.
4. Make a note of your observations and then complete the worksheet on the following page.
5. **Pour all barytes waste into a specially marked beaker on the back counter.** Clean out your test tubes.
6. Repeat steps 1 through 5, but now with nitrate of barytes rather than with muriate of barytes.
7. If time permits, your tutor may permit you to conduct a flame test on one of the precipitates to confirm your reasoning in filling out the worksheet on the following page. To do so, filter the precipitate from the supernatant liquid. When filtering the residue, be sure to rinse the residue with distilled water to make sure that you isolate the residue from the supernatant liquid. Once you have isolated the residue, dip the inoculation loop into the residue and insert the inoculation loop into a Bunsen burner flame. Does the color of the flame match the color of any of the salts that were flame tested above?
8. Clean up your workstation and dispose of all waste in its appropriate place. **All barytes waste goes into a specially marked beaker on the back counter.**

---

[3] In this part, you will be working with several solutions of water and salts. The water is largely inert in the reactions you will be observing.

Worksheet for the Precipitate Reactions

Since precipitate reactions involve one element of a substance swapping places with another in a compound, each reaction necessarily produces two new substances.[4] Fill in the blanks for those word equations where the products are left blank and identify whether the products are soluble or insoluble by circling the appropriate answer. From what you already know (either from your reading or experience in the lab), you should be able to deduce which products are soluble and which are insoluble (i.e., produce a precipitate).

Precipitate Reactions with Muriate of Barytes

Muriate of Barytes	+	Nitrate of Potash	→	**Nitrate of Barytes**	+	**Muriate of Potash**
(soluble)		(soluble)		[✓ soluble] insoluble		[✓ soluble] insoluble
Muriate of Barytes	+	Sulfate of Potash	→	______________	+	______________
(soluble)		(soluble)		soluble insoluble		soluble insoluble
Muriate of Barytes	+	Sulfate of Soda	→	______________	+	______________
(soluble)		(soluble)		soluble insoluble		soluble insoluble
Muriate of Barytes	+	Sulfuric Acid	→	**Sulfate of Barytes**	+	**Muriatic Acid**
(soluble)		(soluble)		soluble insoluble		soluble insoluble

Precipitate Reactions with Nitrate of Barytes

Nitrate of Barytes	+	Nitrate of Potash	→	______________		______________
(soluble)		(soluble)		soluble insoluble		soluble insoluble
Nitrate of Barytes	+	Sulfate of Potash	→	______________	+	______________
(soluble)		(soluble)		soluble insoluble		soluble insoluble
Nitrate of Barytes	+	Sulfate of Soda	→	______________	+	______________
(soluble)		(soluble)		soluble insoluble		soluble insoluble
Nitrate of Barytes	+	Sulfuric Acid	→	______________	+	______________
(soluble)		(soluble)		soluble insoluble		soluble insoluble

---

[4] Since only a few drops of the Barytes are added to each test tube, it is probably safe to assume that all of the Muriate of Barytes and Nitrate of Barytes are changed in the reaction (assuming that there is a reaction). Only a small portion, however, of the four original substances added to the test tubes reacts with the Barytes. So the results of the above equations do not include unreacted Nitrate of Potash, Sulfate of Potash, Sulfate of Soda, and Sulfuric Acid leftover.

Demonstration 6: Metals and Non-Metals Burning in Oxygen

Synopsis

You will observe the combustion of a metal (magnesium) and a non-metal (sulfur) in pure oxygen. Compare the rate and intensity of the burning substance in the oxygen with that in the air. Such a reaction produces what Lavoisier calls an "oxide," meaning a combination of a substance with oxygen. When the metallic and non-metallic oxides are combined with water and tested with litmus paper, we see an important difference between metals and non-metals. The non-metals are capable of being transformed into acids when combined with oxygen and dissolved in water. (This is why Morveau and Lavoisier describe non-metals as "acidifiable bases" in "Method of Chemical Nomenclature.") By contrast, when a metallic oxide reacts with water it produces a base, viz., a metallic hydroxide.

Materials

Magnesium ribbon
Sulfur
Oxygen (red disposable tank)
Oxygen regulator
Tub w/water
4 Erlenmyer flasks
2 Rubber stoppers
Alligator clip attached to metal disc w/handle (for burning magnesium ribbon)
Combustion spoon attached to metal disk (for burning sulfur)
Lighter
Red litmus paper
Red litmus paper

Safety Notice

Burning magnesium produces a bright white light that can damage the eyes. Do not stare directly at the light. Burning sulfur produces a gas that can cause severe lung irritation and should therefore be performed in a fume hood (it also makes it much easier to see the sulfur flame).

Here is the procedure:

1. Fill two Erlenmyer flasks with pure oxygen. Do this by first attaching the oxygen regulator to the red oxygen tank. It threads on by rotating it in the counterclockwise direction. Do not overtighten the regulator. Then submerge an Erlenmeyer flask in the tub of water until it is completely filled with water. Then invert the flask in the tub of water and place the hose coming from the oxygen regulator beneath the mouth of the inverted flask. Turn on the oxygen regulator and fill the flask. When the flask is nearly full, turn off the oxygen and insert a rubber stopper into the mouth of the flask. It is desirable to have a small amount of water in the bottom of the flask, so you should not attempt to fill completely the flask with oxygen. (Note: this step should be performed before class).
2. Add red and blue litmus papers to a flask containing atmospheric air and to one of the flasks containing pure oxygen.
3. Attach a 2" strip of magnesium ribbon to the alligator clip that is attached to the metal disc. Light the end of the magnesium ribbon furthest from where it attaches to the alligator clip

and then quickly insert the magnesium into an Erlenmeyer flask containing atmospheric air so that the metal disk covers the mouth of the flask. Repeat this same procedure with a flask containing pure oxygen. Compare the rate and intensity of the burning substance in pure oxygen with that in atmospheric air.

4. After the combustion is complete, observe any color change in the litmus papers in the jar.
5. Test with litmus papers the liquid produced when distilled water is added to the jar and shaken. Note whether the litmus test results are the same.
6. Repeat steps 1 through 5 with the metal combustion spoon of sulfur.

Report

1. Your report should list the results of your experimental work *(not all* the data, but only what is significant) in as orderly a manner as you can devise, and report the conclusions you derived from your observations. Note: where possible, report your data in a table.
2. What distinctive property or properties did you observe for acids? For bases? In considering the distinctive properties of acids and bases, be sure to consider your observations of the formation acids and bases.
3. What distinctive property or properties of salts did you observe in this experiment? What is the significance of the colors that were observed in the flame test?
4. What are the distinctive properties of metals and non-metals that you observed?
5. In observing the precipitate reactions, did you flame test the precipitates? If so, do your results help you identify the precipitates? Can you write word equations for the precipitate reactions?
6. What hypothesis can you form to explain why the precipitate reactions took place? Do you have any reason to think that the affinity of one particular element for another explains these reactions?

* * * *

Demonstration: The Acidity of Carbonic Acid

In the essays we have read, and again in chapter 5 of the *Elements of Chemistry*, Lavoisier has said that the gaseous oxide produced in the combustion of charcoal, or of carbonaceous substances like wood, is an acid, and has pointed out that this same gas is produced by us when we exhale. But because under ordinary atmospheric pressure it remains as a gas, it is easy to not see its acidity. As Lavoisier notices, however, it can be dissolved in water. Thus, to see that we do in fact have "acid breath," your tutor will have you blow into a beaker of water in which you have placed two pieces of litmus paper, one of each color (why both?), or a drop of liquid litmus, what is usually called an "indicator." After about two minutes of blowing into the water, enough of the carbonic acid gas should be dissolved to change the blue litmus paper red.

Materials

200 mL (or 250 mL) beaker
Drinking straws
Red litmus paper
Red litmus paper

Question

1. How does the acidity of carbonic acid fit with Lavoisier's claim that "all acids are formed by the combustion of certain substances" (See *Atomic Theory Manual*, p. 102)?

Experiment 5: Neutralization of an Acid by a Base via Titration

As we saw in Experiment 4, strong acids and strong bases typically react with one another to produce a neutral salt. Titration is a method used to calculate precisely how much base is needed to neutralize a given amount of acid (or vice versa).

The experiment has a twofold aim:

1) By calculating the precise amount of base required to neutralize a given amount of acid, we can observe the *law of definite proportions*, according to which substances combine in definite and fixed proportions by weight. If 1g of substance A combines with 3 g of substance B, then 2 g of A will combine with 6 g of B, etc. In every case, the proportion A : B :: 1 : 3 remains fixed or constant.[1] If done carefully, titration can yield very precise results illustrating the law of definite proportions.

2) This experiment also enables us to observe Richter's discovery not only that there is a certain definite quantity of base that is "equivalent" to (or neutralizes) a given quantity of acid, but also that there is a certain definite quantity of one base that is "equivalent" to a given quantity of another base. If 36.5 g of muriatic acid (hydrochloric acid) is neutralized by 40.0 g of caustic soda (sodium hydroxide), and is also neutralized by 56.1 g of potash (potassium hydroxide), then 40.0 g of soda can be said to be "equivalent" to 56.1 g of potash. Hence, by calculating the quantities of two different bases required to neutralize a given quantity of acid, one can quantify the amount of the first base that is "equivalent" to the second.

Synopsis

In this experiment we will titrate various concentrations of caustic soda (sodium hydroxide) with muriatic acid (hydrochloric acid). By titrating different concentrations of the same base to a given amount of acid, we can verify that when an acid and a base combine to produce a neutral salt they do so in a definite and fixed proportion by weight regardless of the total amount of acid and base that combine. We will also titrate a solution of potash (potassium hydroxide) with muriatic acid so that we can compare the amount of caustic soda and potash that are equivalent to one another (i.e., neutralize the same quantity of muriatic acid).

Method

Titration makes use of the volumetric pipette and the burette, two measuring instruments that are more precise than those we have used in previous experiments. The volumetric pipette is used to measure an exact quantity of a given solution (e.g., 25 mL). You will be using it to measure an exact quantity of muriatic acid to add to the titration flask. The burette is a precisely graduated glass tube with a stopcock valve on the bottom. Burettes are reverse-graduated—the top is zero and the bottom indicates the total volume. The burette will be used to dispense the base slowly into the flask containing the acid, stopping when we have reached the equivalence point (the neutralization point).

To determine the point at which the solution has reached equivalence, we will use phenolphthalein, which is a pH indicator. It is a colorless acid that turns pink when the solution

---

[1] The law of definite proportions was implied in the chemical analyses of Lavoisier and other early chemists, but the law was first formulated by Richter and made famous by the controversy between Berthollet, who attacked it, and Proust, who defended it.

turns basic. The color change will be used to determine when a solution has reached the equivalence point. Phenolpthalein has already been added to the acid.

Materials

Safety glasses (or goggles)
Gloves
Reactants (note the concentrations indicated by the lab assistant in the spaces below)
  A Muriatic acid (hydrochloric acid) of certain concentration: _____________ g/mL
  B Caustic soda (sodium hydroxide) of certain concentration: _____________ g/mL
  C Caustic soda (sodium hydroxide) of weaker concentration: _____________ g/mL
  D Caustic soda (sodium hydroxide) of weakest concentration: ____________ g/mL
  E Potash (potassium hydroxide) of a concentration: _____________ g/mL
Volumetric pipettes (25 mL and 1 mL)
Pipette filler
Burette and burette funnel
2 small 100 mL beakers, one marked “acid,” the other “base”
250 mL (or 150 mL) Erlenmeyer flask (a conical flask)
200 mL (or 250 mL) beaker marked “waste”
Wash bottle w/distilled water
Clean white paper
Burette reading card (white paper w/black rectangle for reading meniscus)
3-5 Paper towels
2 600 mL beakers for “waste”

Safety Notice

Muriatic acid (hydrochloric acid) is corrosive. Caustic soda (sodium hydroxide) and potash (potassium hydroxide) are caustic. Although the concentrations used in this lab are low, they can cause serious eye damage. Eye protection must be worn at all times.

Titration requires patience and precision to yield useable results. It is very important to follow the instructions closely.

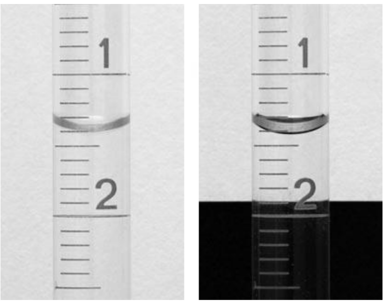
Part I: Clean and adjust the burette and add base solution to the burette

1. To achieve good results, make sure that the burette is clean and in good working order. Begin by carefully removing the burette from the burette clamp. To do this, hold the burette in one hand while squeezing the spring loaded-clamp with the thumb and forefinger of your other hand. If you are unsure how the clamp works, ask your tutor. Glass burettes are very expensive, so treat them with care.
2. To clean the burette, rinse it once or twice with distilled water from the wash bottle. With the stopcock closed, add 3-5 mL of distilled water to the top of the burette. Tilt the burette almost horizontally so that the water runs down the sides (but not out the top). Rotate the burette so that the water wets all surfaces of the glass. Open the stopcock and let some water run out the tip, then close the stopcock and pour the rest out the top and into the lab sink.

3. While rinsing the burette with water, check the burette for leaks. If the burette is leaking from the stopcock valve, try tightening the plastic nut at the end of the stopcock. The stopcock should be tight enough to prevent leaking and loose enough to turn easily. If the burette has a leak that you are unable to fix, talk to your lab tutor before proceeding.
4. Take the two 100 mL beakers, one marked “acid” and one marked “base,” and rinse them with the distilled water from the wash bottle and **dry them thoroughly with paper towel**.
5. Fill the small 100 mL beaker marked “base” with about 75 mL of the base solution assigned to your lab team (note: each lab team will titrate only one of the four bases, viz., B, C, D, or E). Pour carefully and only take the amount indicated by the directions (if you take too much, the other sections will run out). If, however, you happen to pour too much, do not pour from your beaker back into the common bottle! Replace the cap when you are done pouring.
6. Now rinse the burette once or twice with the base solution. To do this, make sure the stopcock is closed and then add 3-5 mL of base using the burette funnel. Tilt the burette almost horizontally so that the solution runs down the sides (but not out the top). Rotate the burette so that the solution wets all surfaces of the glass. Open the stopcock and let some of the solution run out the tip, then close the stopcock and pour the rest out the top and **into the waste beaker**. Install the burette back in the burette clamp.
7. Fill the burette with approximately 50 mL of the base solution so that the solution is just above the zero mark. Again, make sure the stopcock is closed and then use a funnel to add the base to the burette. You may need to lift up on the funnel slightly to allow the solution to flow freely. Remove the funnel. If any solution spills over the sides of the burette wipe it down with a dry paper towel.
8. Remove any air bubbles from the tip of the burette. Place the waste beaker beneath the stopcock and fully open, and then close, the stopcock valve. If you still see air in the tip, try opening and closing the valve rapidly, or tapping the tip of the valve with the stopcock in the open position. If the base solution falls below the zero mark, add more.
9. Carefully and slowly open the burette until the meniscus rests at the zero mark. (See below for instructions on how to read a burette.)

How to Read a Burette

- To avoid parallax error, the level of liquid in a burette must be measured with the meniscus at eye level. The measurement should be taken from where the very bottom of the meniscus rests. Burettes must be read to 0.01 mL which means that the last digit is an estimate.
- To make reading the meniscus easier, it is often helpful to use a burette reading card, a white index card or piece of paper with a black rectangle. If the black rectangle is held about half an inch behind the burette and about half an inch below the meniscus, the edge of the meniscus reflects the color of the rectangle, making it much easier to read.



- Be consistent when reading the burette. The same eye that makes the initial reading should also make the final reading. Any absolute errors will then subtract out. This does not mean that the same lab partner must make all the readings, but the same person should take readings for any one titration.

Part II: Clean the pipette and add 25 mL of muriatic acid to the Erlenmeyer flask

1. Rinse the Erlenmeyer flask two or three times using distilled water from the wash bottle and pour the rinse solution down the drain. It is not necessary to dry the Erlenmeyer flask once you have rinsed it clean. Since distilled water has a neutral pH, any drops of water remaining on the sides of the flask will not interfere with the titration.
2. To save time, the 25 mL volumetric pipette has been pre-rinsed with the acid solution by the lab assistant, so there is no need to rinse the pipette before you begin.
3. Fill the 100 mL beaker marked “acid” with about 30 mL of muriatic acid (hydrochloric acid) from the plastic storage bottle.
4. Using the volumetric pipette, add exactly 25 mL of muriatic acid to the Erlenmeyer flask. Do this by filling the pipette approximately 2-3 centimeters above the graduation line, then remove the pipette filler and quickly position your thumb over the top of the pipette. Slowly decrease pressure applied by your thumb until the meniscus rests on top of the graduation mark. This takes some practice, so you may need to do this 2-3 times before you get the meniscus to fall right on the graduation mark. If you are having trouble getting a good seal between your thumb and the top of the pipette, remove your glove.
5. Dispense the 25 mL of acid into the Erlenmeyer flask. (Note: Do not attempt to blow out the small amount of liquid remaining in the tip of the pipette. That small amount of liquid is supposed to remain in the pipette and the calibration of the instrument takes this into account).
6. When you are finished with the pipette, be place the pipette carefully on the benchtop so that it will not roll off of the counter.
7. Adjust the height of the burette so that the tip is just above the top of the Erlenmeyer flask. You are now ready to begin the titration.

Part III: Titration

1. Begin by placing a white sheet of paper under the Erlenmeyer flask containing the acid so that you will be able to see the color change.
2. Let one person open the stopcock so that a fine stream of the base flows into the flask while your lab partner gently swirls the flask. At this point you may notice pink swirls that immediately disappear.
3. **Watch carefully until you begin to see pink swirls that persist for 2-3 seconds before disappearing.** At this point you should begin adding the base a few drops at a time.

4. **Watch carefully until the pink swirls start to persist for 5-10 seconds.** Record the amount of base for later titrations: ____________ mL and begin adding the base one drop at a time. After each drop, rinse the tip of the burette and sides of the flask with the wash bottle, swirl for a short period of time, and record.
5. Once the entire solution turns a very light pink and stays pink for 30 seconds or more it has reached the equivalence or neutralization point.
6. If you overshoot the neutralization point, you can add 1 mL acid to the solution using a 1 mL volumetric pipette. Make a note of the addition and then repeat steps 4 & 5.
7. Record your results in the table on the following page (and on the blackboard) and make a note of the corresponding results of your fellow students.
8. You should repeat the titration until two successive trials yield volume quotients that agree within one percent.[2] There is no need to rinse the burette before your second (or third) titration.

Cleanup

Neutral solutions can be poured down the drain. Extra acid, or base, should be poured into the waste beakers at the back of the room.

---

[2] For example: suppose 26.00 mL of acid is neutralized by 28.91 mL of soda in a first trial, and 25.0 mL of acid is neutralized by 27.53 mL of soda in a second trial. The volume quotients are 28.91/26.00 = 1.11 and 27.53/25.00 = 1.10. These agree to within 1% of one another.

Team	Trial	Acid		Base		Ratio of Acid to Base by Weight (1 : X)
Team 1 (A:B)	**trial 1**	mL	g	mL	g	
	trial 2	mL	g	mL	g	
	trial 3	mL	g	mL	g	
Team 2 (A:B)	**trial 1**	mL	g	mL	g	
	trial 2	mL	g	mL	g	
	trial 3	mL	g	mL	g	
Team 3 (A:C)	**trial 1**	mL	g	mL	g	
	trial 2	mL	g	mL	g	
	trial 3	mL	g	mL	g	
Team 4 (A:C)	**trial 1**	mL	g	mL	g	
	trial 2	mL	g	mL	g	
	trial 3	mL	g	mL	g	
Team 5 (A:D)	**trial 1**	mL	g	mL	g	
	trial 2	mL	g	mL	g	
	trial 3	mL	g	mL	g	
Team 6 (A:D)	**trial 1**	mL	g	mL	g	
	trial 2	mL	g	mL	g	
	trial 3	mL	g	mL	g	
Team 7 (A:E)	**trial 1**	mL	g	mL	g	
	trial 2	mL	g	mL	g	
	trial 3	mL	g	mL	g	
Team 8 (A:E)	**trial 1**	mL	g	mL	g	
	trial 2	mL	g	mL	g	
	trial 3	mL	g	mL	g	

Calculating the Proportion of Acid to Base

Since pipettes and burettes measure volumes only, and we wish to calculate the ratios of weights, we will have to calculate the latter from the former. This is easily done using the concentrations of the two solutions posted by the lab assistant ("concentration" here being defined as the weight of the substance per unit volume of the solution). For example, A contains .219 g/mL of muriatic acid and B contains .240 g/mL of caustic soda, and then you observe in the titration that 25.00 mL of A is neutralized by exactly 27.53 mL of B. Since the weights must be as the volumes (assuming that each solution is uniform) we can find a fourth proportional:

1.0 mL : 25.00 mL :: .219 g : weight of A used in titration.

Thus, the weight of A = 25.00(.219) = 5.48 g. Likewise, for the base:

1.0 mL : 27.53 mL :: .240 g : weight of B used in titration.

Hence the weight of B = 27.53(.240) = 6.61 g. Thus, we know that 5.48 g of muriatic acid is neutralized by 6.61 g of base. Thus the ratio of acid to base by weight is 1 : 1.21.

Report

1. Present your data and that of your classmates in a table so that you may easily see the results of all of your procedures.
2. Be sure to include a comparison of the ratios by weight of the substances that combine to produce neutral salts.
3. From the information gathered in this lab, come up with a table of equivalence for the three reactants used in this lab.
4. Be sure to include all the possible error factors that might have influenced your results.

Questions and Problems

1. Can your experimental results be used to support the law of definite proportions?
2. What further data would you gather, had you the opportunity, in order to establish (if possible) that a constant composition substance is formed at the point of neutralization?
3. What bearing do the results of this experiment have on the controversy between Berthollet and Proust?

Experiment 6: The Law of Multiple Proportions

Richter's experiments with acids and bases, and Proust's investigations of copper carbonate, led them both to discover that substances tend to combine in fixed proportions. This came to be called the *law of definite proportions*. Lavoisier, however, had already noted the capacity of several substances to combine with oxygen in greater and lesser ratios, and to designate the different degrees of oxygenation he used the suffixes *–ic* and *–ous*. Similarly, Berthollet and other chemists noticed that acids and basis do not always form neutral salts but in some cases form salts that are acidic or alkaline. These observations led Dalton and Wollaston to modify, or extend, the law of definite proportions.

According to Dalton's doctrine of atoms, if two elements form more than one compound, then the ratios of the quantities of the second element that combine with a fixed quantity of the first element will be in ratios of small whole numbers. They will be in ratios of whole *numbers* because the various amounts of the second element that combine with a fixed amount of the first represent whole numbers of atoms; and *small* because according to Dalton the most prevalent combinations may be presumed to be those between the least numbers of atoms. Wollaston called this the *law of multiple proportions*. In this experiment you will attempt to verify this law.

Synopsis

Copper unites with oxygen in two different proportions to form two distinct oxides, cuprous oxide (red copper oxide) and cupric oxide (black copper oxide). We will reduce each oxide to elemental copper by heating it in a stream of burner gas (propane). By establishing the weight of metallic copper obtainable from a given amount of each oxide, we can calculate the proportions between copper and oxygen in the two oxides. On Dalton's hypothesis, if the quantity of copper is fixed at 1 gram, the two amounts of oxygen should be to one another in the ratio of small whole numbers.

Materials

Cuprous oxide (red copper oxide)[1]
Cupric oxide (black copper oxide)[1]
Large test tube (25x200mm)
#4 solid rubber stopper
Bunsen burner
Clamp stand, two-prong utility clamp
One-hole rubber stopper with glass and latex tubing attached
Scoopula
Weigh paper
Digital scale with 0.001g precision
Gloves, Safety glasses
Large test tube brush

---

1. Red and black copper oxides are hygroscopic (i.e., they tend to absorb water) and therefore must be dried thoroughly before this experiment. Any water absorbed by the copper oxides will distort the weighing and lead to bad results. The oxides should be placed in a drying oven at 150°C for 6-8 hours. After drying, they can be stored at a lower temperature in a desiccator with a drying agent.

Safety Notice

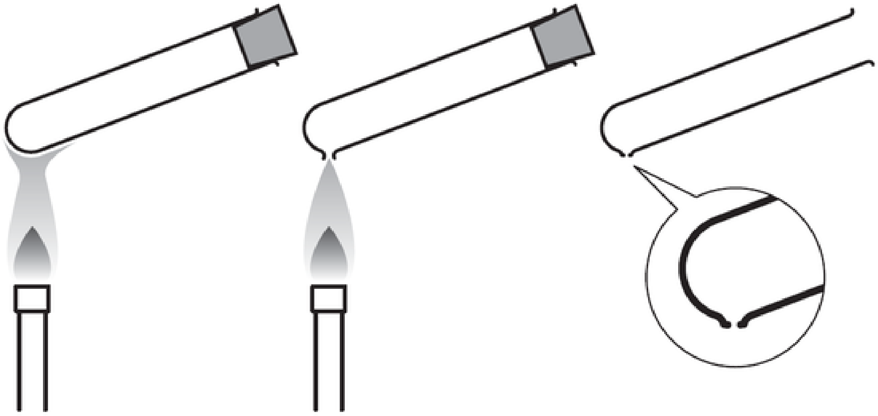
During the first few minutes of this experiment a combustible gas-air mixture forms in the apparatus, presenting a danger of explosion if accidentally exposed to flame. Therefore adhere carefully to the waiting period outlined in step 8 of Part II. All members of the class should wear safety glasses.

Red and black copper oxides are poisonous if inhaled, ingested, or absorbed through the skin. Avoid dispersing dust and wash your hands thoroughly if you get it on your skin.

Part I: Making a Reaction Tube

Heating copper oxide in a stream of burner gas requires making a reaction tube, a modified test tube or boiling tube. Heating a borosilicate test tube fitted with a rubber stopper causes the pressure inside the tube to rise and the glass to soften. After a few minutes the test tube will "pop." Here is the procedure:

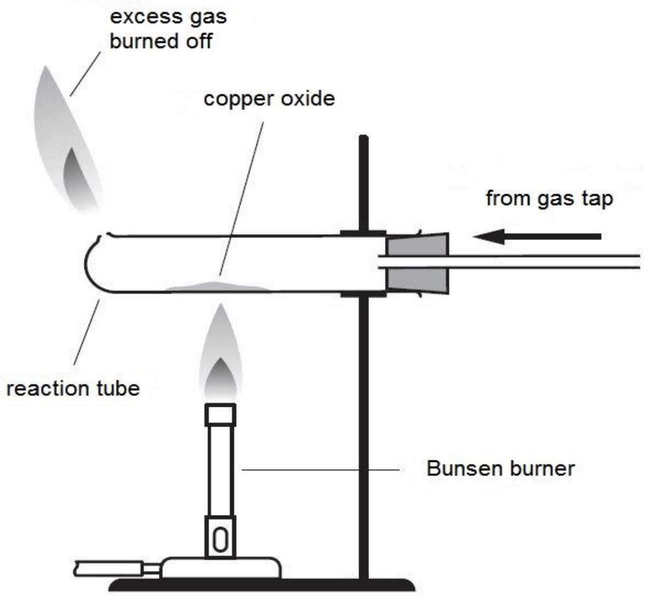
1. Insert the rubber stopper firmly into the test tube.
2. Light the Bunsen burner and adjust the barrel so that it produces a hot flame with a well-defined inner blue cone. At this point you should be wearing safety glasses.
3. Holding the top of the tube by hand, insert the bottom of the tube in the hottest part of the flame as indicated in the diagram below. Heat a single spot. Do not rotate the tube. (Note: Holding the test tube by hand is perfectly safe because glass is a poor conductor of heat along the length of the tube. If you are nervous about burning yourself, you can clamp the boiling tube in the two prong clamp rather than hold it by hand. The clamp should be snug, but not overly tight).
4. The glass will begin to deform and then "pop."
5. Heat the glass around the hole for a few seconds. The sharp edges of the glass will form a nice neat hole.
6. Remove the tube from the flame and let it cool for several minutes. Be careful not to touch the bottom of the tube until you are confident that the glass is cool.



Part II: Reducing Copper Oxide with a Stream of Burner Gas

In the first semester we observed the reduction of copper oxide by heating it in the presence of carbon thus producing carbonic acid gas. In this lab we will reduce copper oxide by heating it in a stream of ordinary burner gas, a compound of carbon and hydrogen. When heated together, the copper oxide and burner gas decompose and the oxygen from the copper oxide combines with both the carbon and the hydrogen from the gas to produce carbonic acid gas and water vapor, both of which escape through the outlet hole in the reaction tube. This leaves elemental copper. Here is the procedure:

1. Carefully weigh the reaction tube to the nearest 0.001g. Reaction Tube: ___________ g.
2. Using a piece of weigh paper, weigh approximately 1g of red copper oxide.
3. Carefully transfer the oxide into the reaction tube so that it is evenly distributed in a line about 3 inches long opposite the small hole you produced in Part I. The best way to do this is as follows: a) carefully pour the red copper oxide into the blunt end of a scoopula so that it is evenly distributed in the last 3 inches of the scoopula, b) holding the tube horizontally, insert the scoopula into the reaction tube until it reaches the desired position, and c) rotate the scoopula until the copper oxide drops out of the scoopula.
4. Weigh the reaction tube again with the copper oxide. Tube + Copper Oxide: ___________ g.
5. Calculate the weight of the copper oxide to the nearest 0.001g. Copper Oxide: __________ g.
6. Carefully clamp the reaction tube to the clamp stand using the two-prong utility clamp. Do not overtighten. The clamp should be tight enough to hold the test tube in place, but loose enough that the test tube can be moved from side to side.
7. Insert the rubber stopper with attached surgical tubing into the end of the reaction tube and connect the other end of the surgical tubing to a gas tap. The apparatus should look like this:



8. Turn on the gas tap so that a stream of gas flows through the reaction tube. **Note that we are introducing burner gas into glassware that originally contained air. Eventually the incoming gas will displace all the air from the apparatus, but until that time the tube will contain a combustible mixture of gas and air. Allow the gas to flow vigorously for a full 30 seconds. This length of time is sufficient for burner gas to replace all of the air in the tube.**
9. After 30 seconds, partially close the gas valve so that a more moderate stream of gas is emitted from the small hole at the end of the reaction tube, and then carefully light the stream of gas.
10. Adjust the flame coming from the end of the tube so that it is roughly 1½ inches tall.
11. Light the Bunsen burner and adjust the height of the reaction tube so that the bottom of the reaction tube is in the hottest part of the flame.
12. Begin heating the copper oxide. You will notice that the red copper oxide will begin to change color, first turning black and then turning copper pink.
13. Continue heating for roughly ten minutes until you are confident that all of the oxide is reduced. You may need to move the burner so that all of the oxide in the tube is heated sufficiently. Be careful, however, not to melt the rubber stopper!
14. Once you are confident that the oxide has been fully reduced to copper metal, turn off the Bunsen burner. **Do not turn off the stream of gas flowing through the reaction tube! If you turn off the gas before the apparatus cools, the copper in the reaction tube will re-oxidize and you will have to go back to step 8.**
15. Let the reaction tube cool to room temperature in a current of burner gas, and only then shut off the gas supply. To be safe, let the reaction tube cool for approximately ten minutes.
16. Once the tube is completely cool, disconnect the stopper and tubing from the reaction tube and weigh the tube and its contents again to the nearest 0.001g.

    Tube + Copper: _____________ g.
17. Calculate the weight of the copper recovered from the reduction. Copper: _____________ g.
18. Calculate the weight of the oxygen lost during the reduction. Oxygen: _____________ g.
19. Record your results on the blackboard and in the table on the following page. Make a note of the corresponding results of your fellow students.
20. Using a clean scoopula and dry test tube brush, clean out the copper from the reaction tube. Put the copper into the beaker marked “copper waste.” **Do not use water!** Do not worry if you are unable to remove all traces of the copper.
21. Repeat the procedure for black copper oxide starting with step 1. (Yes, you do need to weigh the reaction tube again! If there are small amounts of copper left in the tube it will weigh more than the first time.)

Team	**Red Copper Oxide**				
	Reaction Tube	**Oxide**	**Copper**	**Oxygen**	**Copper:Oxygen (1:x)**
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
Class Average	▨	▨	▨	▨	
	Black Copper Oxide				
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
Class Average	▨	▨	▨	▨	

Part III: Calculating the Combining Proportions of Copper and Oxygen.

In Part II we calculated the actual weight of copper and oxygen that combine in the two oxides. All that is left to do is to simplify the two proportions by determining how much oxygen would combine with one gram of copper to form the two oxides. Once we do so, we can more easily see whether the experimental data support the law of multiple proportions. Here is a hypothetical example:

> If 1.075g of red copper oxide loses 0.121g of oxygen, leaving 0.954g of copper, then the proportion of copper to oxygen is 0.954 : 0.121. We can simplify the proportion by setting copper equal to one and finding the fourth proportional. If 0.954 : 0.121 :: 1 : x, then x = 0.127. The combing proportion of copper to oxygen is 1 : 0.127.
>
> If 1.129g of black copper oxide loses 0.234g of oxygen, leaving 0.895g of copper, then the proportion of copper to oxygen is 0.895 : 0.234. Again, we can simplify the proportion by setting copper equal to one and finding a fourth proportional. If 0.895 : 0.234 :: 1 : x, then x = 0.261. The combining proportion of copper to oxygen is 1 : 0.261.
>
> If we then compare the two proportions we see that the quantity of oxygen in the two oxides of copper are in the ratio of 0.127 : 0.261, which is approximately the relation of 1 : 2, a simple whole number ratio.

Report

1. Present your data and that of your classmates in a table so that you may easily see the results of all of your procedures.
2. Be sure to include the calculation of the class average of the ratio of copper to oxygen in the two copper oxides.
3. Examine the relationship between the average results, and consider, on the assumption that the copper at the end of the reduction is the same for both the red and black copper oxide, whether your data suggests that the amount of oxygen in one is a simple multiple of the amount of oxygen in the other. It may be helpful to compare Wollaston’s equivalents for Black Copper Oxide and Oxygen (viz., 50 and 10); see pp. 127-129. You may also make this comparison using modern values for these equivalents (viz., 79.5 and 16); we will come across these values later in the manual. Is the law of multiple proportions, as stated in Wollaston, verified by your results?
4. List all the possible error factors that should be accounted for in your results.

Demonstration 8: Two Kinds of Electricity

Two glass rods will be rubbed with a piece of silk. Two hard rubber rods will be rubbed with a piece of fur. These four rods, freely suspended, will be brought into proximity of each other. Note what happens.

Demonstration 9: The Electrolysis of Water and Gay-Lussac's Law

Using the process of electrolysis, Humphry Davy discovered six new elements (sodium, potassium, calcium, magnesium, strontium, barium). Electrolysis can also be used to decompose water into its constituent elements, hydrogen and oxygen. Since hydrogen and oxygen are both gases at room temperature, the electrolysis of water can also be used to verify Gay-Lussac's Law of Combining volumes—that substances combine in simple whole number ratios by volume.

Method

The Hofmann Voltameter is the apparatus used to demonstrate that electrically conductive liquids can be decomposed and resolved into their constituent elements by means of unidirectional electric current. The gaseous components thus formed are trapped, and their relative volumes are measured.

Materials

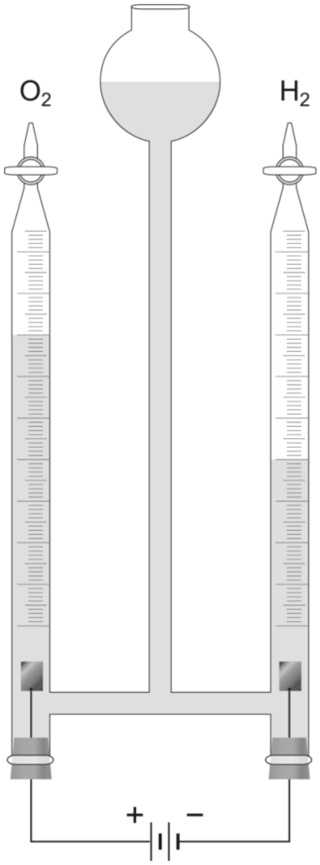
Hofmann Voltameter
DC Power supply
Wire test leads with alligator clips
Ring stand
2 rubber coated utility clamps
Lighter
Wooden splints
Test tubes
4-5 inch length of vinyl tubing
Distilled water
Concentrated sulfuric acid
Goggles
Butyl gloves (black colored gloves)

Safety Notice

The setup of the Hofmann Voltameter involves handling concentrated sulfuric acid which is highly corrosive. The tutor or lab assistant setting up the apparatus should be wearing goggles, butyl gloves, and a lab apron. Once the apparatus is set up, these precautions are unnecessary.

Description of the Hofmann Voltameter

The assembled Hofmann electrolysis apparatus is show in the illustration on the following page. The glassware unit consists of three vertical tubes joined to a cross-tube near the bottom. Two tubes are of equal length and are graduated. The third tube, located between the others, is longer and is topped by a globular reservoir. Affixed to the top of each graduated tube is a stopcock. Graduations extend from the stopcocks downward to the 60ml marks in 0.2 ml divisions. The base of each graduated tube is flared and tapered to accommodate a rubber stopper, on which is mounted a platinum electrode assembly. Each electrode assembly consists of platinum electrode connected to a wire lead sealed within a glass tube, which passes through the rubber stopper. Lead wires protruding from the sealed glass tube connect to the terminal posts on the base.



Setup Procedure (before class)

1. Insert the electrodes in the open, tapered end of each graduated glass tube. Carefully press upward on the rubber stoppers to insure tight closures.
2. Clamp the Hofmann apparatus to a ring stand using two utility clamps so that it is raised approximately 6 inches off the table top. **Do not overtighten the clamps!**
3. Fill the apparatus about half-way with distilled water. Then slowly add approximately 20 mL of concentrated Sulfuric Acid. Then fill the rest of the apparatus with distilled water so that the water level comes to the bottom of the reservoir. To fill the side arms with the acidulated water, turn the stopcocks to the “open” position to allow the atmospheric air to escape the system.
4. After the apparatus is filled with water, connect the wire leads to the electrodes at the bases of the rubber stoppers and to the DC power supply. The power supply should be set to 12V DC power.

Electrolysis

1. Turn the power source on. The anode (positive) side produces oxygen and the cathode (negative) side produces hydrogen. They should be in 1 : 2 ratio by volume.
2. To verify that the two gases are hydrogen and oxygen, test the gases using a wooden splint.

3. Test for oxygen gas as follows. Collect some of the gas by attaching a small 4-5" length of vinyl tubing to the top of the stopcock, bending the vinyl tubing so that it is faces down into a small test tube. (When collecting oxygen, the mouth of the test tube should be facing up because oxygen is heavier than atmospheric air.) Open the stopcock to collect the gas. Cover the mouth of the test tube with your thumb while an assistant lights a wooden splint. Blow out the flame, and insert the glowing end of splint into the test tube. The oxygen should make the splint glow brighter.
4. Test for hydrogen as follows. Collect some of the gas by holding an inverted test tube over the end of the stopcock. (When collecting hydrogen, the mouth of the test tube should be facing down because hydrogen is lighter than atmospheric air.) Open the stopcock to collect the gas. Cover the mouth of the test tube with your thumb while an assistant lights a wooden splint. Insert the flaming end of the splint into the mouth of the test tube. The hydrogen should produce a squeaky pop sound.

Discussion Questions

1. Where in the apparatus would we suppose the water is being decomposed?
2. Based upon your observations, what can you conclude about the electrical nature of hydrogen and oxygen gas? Water?
3. A substance that migrates to, or is neutralized at, a negative pole is considered to be an electrically positive (**electropositive**) substance. Why? A substance that migrates to or is neutralized at a positive pole is considered to be an electrically negative (**electronegative**) substance.
4. Do volumes of gas produced illustrate Gay Lussac's Law of Combining Volumes? Try to explain any deviation from the prediction based on the law.
5. At the beginning of the 19th century, Davy employed the technique of electrolysis via the voltaic pile with great success. In theorizing about what was happening, he proposed that chemical changes and electrical phenomena

> are conceived to be *distinct* phenomena; but produced by the *same power*, acting in one case [electrical phenomena] on masses, in the other case [chemical changes] on particles. . . The same arrangements of matter, or the same attractive powers, which place bodies in the relations of positive and negative, i.e., which render them attractive of each other electrically . . . may likewise render their particles attractive, and enable them to combine, when they have full freedom of motion. . . . It has been supposed that the idea was entertained that chemical changes were occasioned by electrical changes—than which nothing is further from the hypothesis which I have ventured to advance.[2]

Does Davy's position about the relationship between electrical phenomena and chemical combination and decomposition seem plausible, given our electrolysis experiment?

---

[2] *Elements of Chemical Philosophy*, 1812, p. 162, quoted in J. R. Partington, *A Short History of Chemistry* (Macmillan, 1960), p. 191.

Demonstration 10: Reactive Metals

The electrolysis apparatus that we use works only for liquids that decompose into gases, so it will not allow us to replicate Davy's experiments. We will instead perform something like their converse, by observing the reactions of elemental sodium and potassium with distilled water.

Materials

Sodium metal
Potassium metal
Distilled water
Scoopula or Knife
Chem rock
Ring stand and utility clamp
2 large test tubes (25x200mm)
Glass bowl (pyrex)
Detergent
Lighter
Wooden splints
Litmus paper (or phenothelein)
Gloves and safety glasses (or goggles)

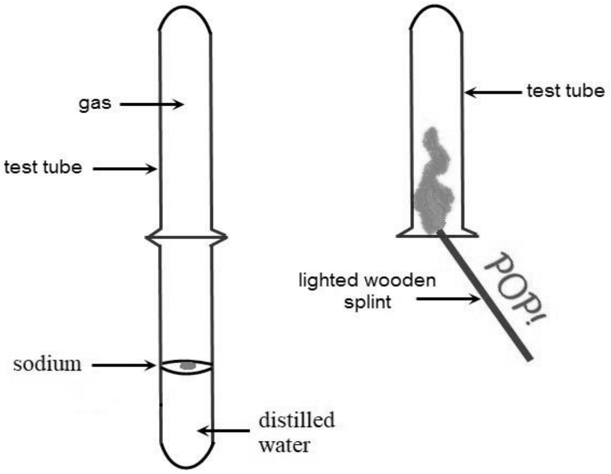
Safety Notice

Always wear gloves when handling reactive metals as the moisture on one's fingers could easily ignite the metals, and because even a little bit caught under one's nails could later result in injury. All those observing the demonstration should be wearing safety glasses (or goggles).

Part I: Sodium

1. Make sure your gloves, the scoopula (or knife), and the Chem Rock are all dry before you handle and cut the metals.
2. Begin by removing a lump of sodium from the storage container (the metal is typically stored in mineral oil so that it cannot react with moisture in the air). Using the pointed end of the scoopula (or knife), stab a lump of sodium and remove it from the storage container.
3. Place the lump on the Chem Rock and, using the scoopula (or knife), cut off a piece of sodium about the size of a marble. Return the rest of the sodium to the storage container.
4. Allow the students to observe the properties of the sodium. Note how easy it is to cut and how malleable it is. Not also the metallic luster when it is freshly cut, as opposed to the dull gray color of the metal which is oxidized on the exterior surface of the lump. Note also how quickly the metal reacts with the air, losing its luster.
5. Cut off a tiny piece of sodium about the size of a peppercorn, and roll it into a ball so that everyone can see how malleable it is.
6. Drop the piece of sodium into a large test tube that is clamped to a ring stand and filled about ¼ full with distilled water. Observe the reaction.

7. Notice the effervescence of a gas. Collect some of the gas by holding an inverted test tube over the mouth of the test tube containing the sodium, then insert a lighted wooden splint into the mouth of the inverted test tube. You should hear the squeaky pop that is characteristic of hydrogen.



8. Since the reaction of sodium and water is exothermic, you should be able to observe the change in temperature by feeling the outside of the test tube. Wait until the sodium has finished reacting.
9. Next, fill a pyrex glass bowl about 2/3 full of distilled water. Add a drop of detergent to prevent the metal from sticking to the side. Before adding the metal, note the temperature of the water using a thermometer.
10. Cut a piece of sodium about the size of a pea, and drop it into the water. Observe how the reaction is more vigorous due to the larger surface area of the sodium. Note also that the sodium floats on the surface of the water. This is because the sodium is less dense than the water and because the hydrogen produced by the reaction provides a cushion between the metal and the surface of the water.
11. If the sodium catches fire, note the orange yellow flame. You should recognize this color from the flame tests we conducted in Experiment 4.
12. Test the water left in the bowl using litmus paper (or alternate indicator).
13. When you are finished, pour the water from the test tube and the pyrex bowl down the drain with copious amounts of water.

Part II: Potassium

1. Repeating the procedure above, removing a lump of potassium from the storage container and cut into it so that students can observe how its properties are similar to those of sodium. It is malleable like the sodium and produces a more volatile reaction.

2. Cut a very small piece of potassium about the size of a grain of rice, and drop it into a pyrex glass bowl that is 2/3 filled with distilled water. Since potassium is more volatile than sodium, this part of the demonstration should be carried out under the fume hood.
3. As the potassium catches fire, note the lilac flame. You should recognize this color from the flame tests we conducted in Experiment 4.
4. Test the water with litmus paper after the reaction and compare your results with your sodium results.
5. When you are finished, pour the water from the test tube and the pyrex bowl down the drain with copious amounts of water.

Questions

1. How would you classify chemical reactions that take place when the metals react with water? Can you write word equations for these reactions?
2. How do these reactions fit with the electrochemical theory of Berzelius?