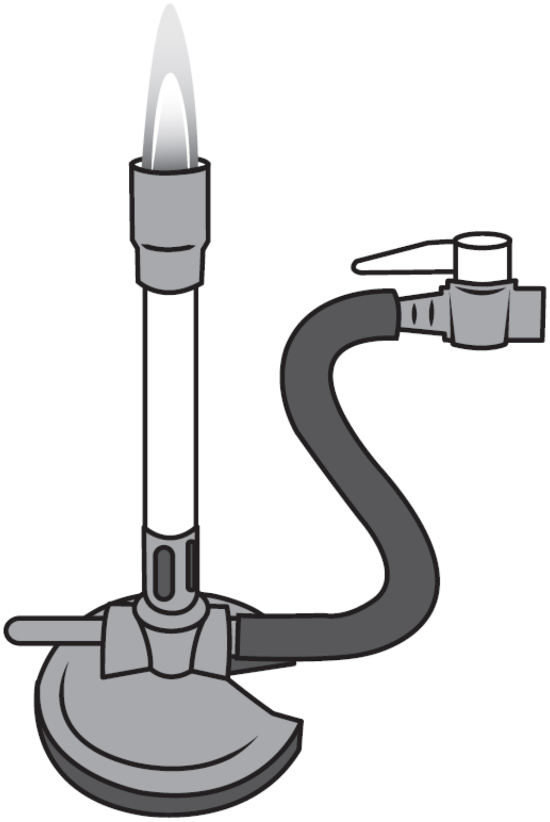

VALENCIA COLLEGE

GENERAL CHEMISTRY 1 LABORATORY

WEST CAMPUS AND LAKE NONA CAMPUS

3RD EDITION REV A

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VALENCIA COLLEGE

Experiment 1:
Mass, Volume, and Density

Version 3a

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In this experiment, you will use common glassware and equipment in order to study the physical property of density.

Objectives

- Recognize and utilize common glassware and equipment.
- Prepare solutions of different densities to identify unknowns.

Learning Outcomes

- Understand the nature of units of measurement and apply proper significant figure rules.
- Differentiate between accuracy and precision.
- Understand the nature of matter and its underlying physical and chemical characteristics.
- Employ conceptual learning outcomes and perform essential lab techniques in a laboratory setting.

Definitions

- **Accuracy –** refers to how close the measured value is to the true, correct, or accepted value
- **Balance –** an instrument to measure mass precisely
- **Buret –** a long glass tube with graduations and a stopcock for dispensing variable amounts of liquid, especially in titration
- **Certain digits –** for an analog instrument, they are unambiguous digits clearly indicated by the instruments; for a digital instrument, they are all digits displayed by the instrument except for the one furthest to the right
- **Concentration –** the amount of dissolved material in a unit of volume
- **Condition –** to rinse a piece of glassware with the solution to be contained, so that all contaminants are removed
- **Density –** the degree of compactness of a material; defined as the ratio of mass to volume, often reported in terms of g/mL or g/cm^3
- **Graduated Cylinder –** a narrow cylinder with graduations to measure volume
- **Homogeneous –** having uniform composition and properties throughout
- **Mean –** the arithmetic average of a set of values

- **Meniscus –** the curved shape of the top surface of a liquid in a cylinder or tube; it should be read at the bottom of the concave shape for most liquids although some such as mercury have a convex shape (read the top of the meniscus when it is convex)
- **Plastic –** a broad descriptor for a range of natural and synthetic materials that can be molded into shapes; they typically have high molecular mass components and are carbon-based
- **Precision –** refers to how close a series of measurements are to one another
- **Relative percent error –** a calculation that indicates the accuracy of an experimentally determined value against the true, correct, or accepted value
- **Significant figures** (or **significant digits) –** all the certain and uncertain digits in a measurement; they should be recorded and carried through calculations since the calculated value must reflect the precision of the measurements
- **Solute –** the substance dissolved in solvent in a solution
- **Solution –** a homogeneous mixture
- **Solvent** – the major component of a solution
- **Standard deviation –** a calculation that indicates the precision of a series of values by showing the variation around the average or mean
- **Tare –** the mass of an empty container; a balance with an empty container can be set to zero so that only materials added will be read in the mass
- **Uncertain digit –** the last digit of a measurement; it is always estimated, even in a digital instrument, and based upon the reliability of the instrument
- **Volumetric flask –** a long-neck flask for measuring one volume with great certainty
- **Volumetric pipet –** a pipet for measuring one volume only with great certainty
- **Weighing boat** (or **weighing paper) –** a container used for weighing samples

Techniques

- Technique 1: Cleaning Glassware
- Technique 2: Using a Balance
- Technique 3: Transferring Liquids
- Technique 4: Using a Graduated Cylinder
- Technique 5 Video Tech. 5: Solutions Using a Volumetric Pipet
- Technique 6: Solutions Using a Volumetric Flask
- Technique 11: Disposing Chemical Waste

Introduction

Figure 1. Plastic Debris on Hawaii's Big Island. Image from Daniel Cressey, Nature Publishing Group.

Plastics are incredibly versatile materials that have made much of modern life possible. Advocates highlight their use in everything from clothing, building materials, medical devices, and packaging to preserve food or allow aseptic technique.[1,2] They are lightweight compared to steel and glass, and so have decreased carbon dioxide emissions when shipping and when used to build vehicles. There are downsides to plastic too: often it is made from petroleum, and it can be disposed inappropriately, leading to extra waste in landfills and pollution in the environment such as on beaches (Figure 1)[3,4] and in the ocean such as the Great Pacific Garbage Patch.[5]

Although there is great interest in recycling, there is not enough recycled plastic for big companies to have a steady supply; some people still throw plastic into the waste bin and many cities do not have comprehensive recycling programs.[6] Currently, several major companies including Walmart® and Coca-Cola® have created the $100 million Closed Loop Foundation to give interest-free loans to cities and recycling companies to improve their recycling.

To recycle, scrap plastics are sorted per resin identification code (Table 1); usually, you can see the code inside the triangle symbols on the material. Once sorted, the plastic is cut into chips or flakes, washed, and then melted into pellets for reprocessing. If the wrong plastics are mixed, they can ruin a batch.

> *In a world of nearly seven billion souls and counting, we are not going to feed, clothe and house ourselves solely from wood, ore and stone; we need plastics. And in an era when we're concerned about our carbon footprint, we can appreciate that lightweight plastics take less energy to produce and transport than many other materials. Plastics also make possible green technology like solar panels and lighter cars and planes that burn less fuel. These "unnatural" synthetics, intelligently deployed, could turn out be nature's best ally.*
>
> *-Susan Freinkel*

To introduce the experiments for this laboratory, you will have a scenario to demonstrate a potential application of the chemical principles that you are learning. Today, you will play the role of technician at the I've Bin Recycling Company. The optical sorter at the Materials Recovery Center, which relies on infrared spectroscopy to separate the different plastics,[7] has failed. A batch of plastics has been made into chips from accidentally mixed plastics. You have been tasked to find a quick and easy way of separating the different plastic chips and objects until the optical sorter is fixed. By remembering a little bit of your undergraduate general chemistry, you can devise a system based upon densities.

To be an effective technician, you will need to record your observations and data to the appropriate significant figures based upon reliability of the instruments. Several instruments can be used to make the same measurement, but the accuracy of the measurement may be different. While approximate measurements are acceptable in some cases, you often will need to measure accurately.

Table 1. The Resin Identification Codes[a] to Recycle Plastics and Examples of Common Usage.

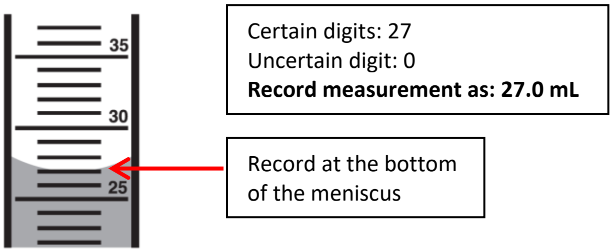
Type of Plastic	It starts as:	It is recycled into:
1 PET **Polyethylene Terephthalate**	Water and soda bottles, jars, clamshells	Carpeting, tennis balls, water and soda bottles, clamshells
2 HDPE **High-Density Polyethylene**	Grocery bags, juice and milk bottles, detergent and shampoo bottles	Plastic lumber, trash cans, toys
3 PVC **Polyvinyl chloride**	Cleaning supply jugs, pipes, pool lining, sheeting	Pipe, floor mats, computer cords
4 LDPE **Low-Density Polyethylene**	Food storage containers, squeeze bottles, trash bags, dry-cleaning bags, six-pack rings	Toys, lawn furniture, trash bags, shipping envelopes
5 PP **Polypropylene**	Medicine bottles, yogurt containers, straws, hangers	Brooms, toothbrushes, speed bumps, flower pots, auto parts
6 PS **Polystyrene**	Vitamin bottles, to-go containers, hot cups, CD cases, cartons	Building insulation, food service trays, picture frames
7 Other **Other Plastics**	Acrylic, nylon, polycarbonate, PLA (polylactic acid/corn plastic), ABS (acrylonitrile butadiene styrene, found in Legos®)	Electronic housings, auto parts, pens, street signs

[a] Note that the Resin Identification Codes were updated from chasing arrows to triangles in 2013 by the American Section of the International Association for Testing Materials (ASTM International).[8]

Making and Recording Measurements

In a laboratory, the precision and accuracy of results are limited by both the instrument, and user proficiency for reading the instrument correctly. In this laboratory, you will use several instruments for measuring the volume of a liquid. Even the best instrument has some uncertainty associated with it. To properly record a measurement, read all digits that are certain plus the first uncertain digit. Both the certain digits and one uncertain digit are significant figures (or significant digits).

In a graduated cylinder, the measured liquid creates a meniscus, a curved shape in a cylinder; read from the bottom of the meniscus for most solutions. You can read all the indicated graduations plus make one estimate for the location of the bottom of the meniscus. For example, in Figure 2, notice graduations of 25 and 30 mL on the graduated cylinder. Now notice that there are four smaller markings between the two stated numbers, each representing 1 mL. The liquid is clearly on the 27-mL line, so those are certain digits. In addition, you can estimate one more digit between the graduations. Since the meniscus is on the line, record the volume as 27.0 mL. Make sure to view the graduated cylinder with your eye level to the meniscus, and the cylinder on an even surface.

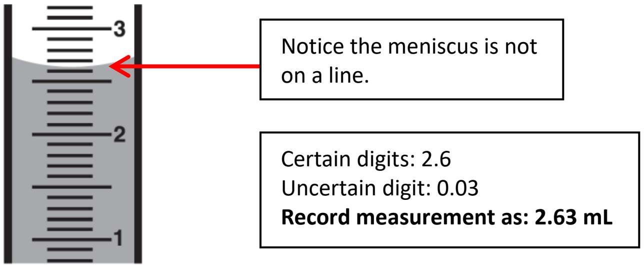


Certain digits: 27
Uncertain digit: 0
Record measurement as: 27.0 mL

Record at the bottom of the meniscus

Figure 2. Reading a Graduated Cylinder.

In a second example, shown in Figure 3, notice the graduations at 2 and 3 mL with nine markings between them, each corresponding to 0.1 mL. In addition, notice how this meniscus is not on a line but rather between 2.6 and 2.7 mL. The certain digits in this volume will be 2.6 mL. Using your best judgment, approximate how close the meniscus is to the next marking. Since the meniscus appears to be less than halfway between the sixth and seventh marking, the uncertain digit might be estimated to be 0.03 mL (i.e., to the hundredths place). Therefore, the volume would be recorded as 2.63 mL. Two people may make different estimates on this last digit, hence the uncertainty.

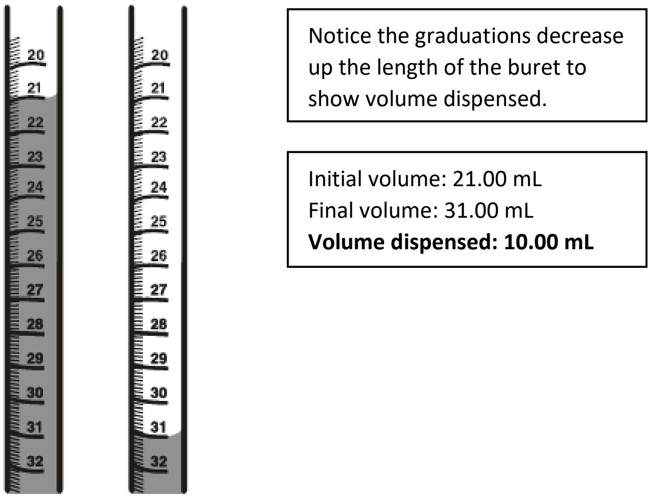


Notice the meniscus is not on a line.

Certain digits: 2.6
Uncertain digit: 0.03
Record measurement as: 2.63 mL

Figure 3. Estimating on a Graduated Cylinder.

When measuring, note carefully the units and graduations of each instrument. The graduations in a buret run in the opposite direction of the graduated cylinder since they measure liquid dispensed (Figure 4).



Notice the graduations decrease up the length of the buret to show volume dispensed.

Initial volume: 21.00 mL
Final volume: 31.00 mL
Volume dispensed: 10.00 mL

Figure 4. Reading a Buret.

To be more precise when measuring volume, you can use volumetric glassware - pipets or flasks (Figure 5). Volumetric pipets measure one volume only, with more certainty than a graduated cylinder. The tolerance of the glassware, which describes its accuracy, is indicated near the label, so a 1-mL pipet with tolerance of ±0.006 mL would be sure to measure between 0.994 mL and 1.006 mL; its volume is recorded as 1.000 mL. It is the microliter (the third decimal position) that is the first uncertain digit. If the tolerance is not listed on the glassware, assume it is accurate to ±0.01 mL. Laboratory Techniques 5 and 6 describe the steps for use of volumetric flasks and pipets.

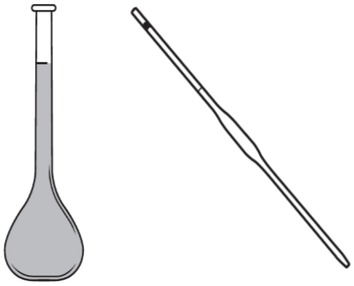


Figure 5. Volumetric Flask (left) and Pipet (right).

For digital or electronic measurements, such as mass on an analytical balance (Figure 6), record all digits as significant. The uncertain digit is the farthest to the right; you may notice it fluctuates a little as you make your readings. Laboratory Technique 2 describes the steps for measuring on the balance.

Figure 6. Analytical Balance.

Again, certain and uncertain digits are all significant. Increasing significant figures increases the certainty or precision of the measurement. The technique of recording all certain digits and the uncertain digit must be performed actively in the lab.

Density

Matter has mass and occupies space. The relationship between these two characteristics is called density; it is a measure of the compactness of matter. Since density is a physical property, it can be used to determine the identity of a substance, as you will do today. To calculate density (d), divide the mass (m) of the object by its volume (V). The most common units for density in chemistry are g/cm^3 for solids, g/mL for liquids, and g/L for gases.

$$d = \frac{m}{V}$$

For practice, you will measure known volumes and determine the density of water with both a volumetric pipet and a graduated cylinder. The interest is not only in determining the density of water, but also to check the precision and accuracy of the instruments - and your skills - by calculating relative percent error (rel. % error) and standard deviation. First, make several measurements and take the mean ($\bar{x}$) to minimize random errors (errors that are unpredictable). For three trials, the mean is:

$$\bar{x} = \frac{trial_1 + trial_2 + trial_3}{n}$$

where *n* is the number of trials.

Relative percent error will demonstrate how accurate a measurement is by comparing it to the true or accepted value. Since density is temperature- and pressure-dependent, use the density value based on the water temperature and barometric pressure from Appendix 1 as the accepted value. The formula for relative percent error is:

$$relative\ \%\ error = \left|\frac{true\ value - calculated\ value}{true\ value}\right| \times 100\%$$

Standard deviation will demonstrate precision by showing the variation in a set of values around the mean. The formula for standard deviation (s) is:

$$s = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n-1}}$$

where x_i is each value in the set of measurements, $\bar{x}$ is the average or mean of the set of measurements, Σ is the sum, and n is the total number of measurements in the set of measurements. Standard deviation has the same unit of measure as the average, and should be reported to the same number of decimal places as the average (subtraction rule).

Scenario

For the I've Bin Recycling Company, you will determine the identity of your unknown plastic flakes and objects by density. You can measure object masses on the balance. For volume, you will find that some objects have regular sides, some are irregular, and some float on water, so you will need to find a protocol to use for each. Following are some suggested techniques for measuring volume.

Regular-shaped Object

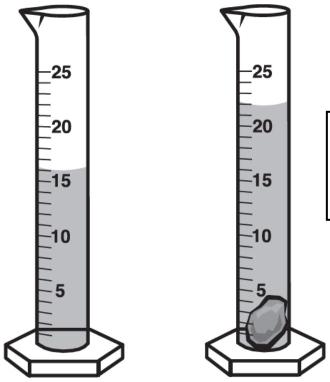
For a regular-shaped object, you might be able to measure its volume with a ruler. The formula for a rectangular solid is:

$$V = l \times h \times w$$

where l is length, h is height, and w is width for the object.

Irregular-shaped Object

For an irregular object, you might choose to measure its volume by water displacement (Figure 7) if it can be submerged in water. An initial volume of water is measured. Then, the object is added, and the new volume is measured. The difference in volumes corresponds to the volume of the object.



Initial volume: 16.0 mL
Final volume: 22.0 mL
Volume of rock: 6.0 mL

Figure 7. Measuring Volume by Water Displacement.

For an object that floats on water or is too small to measure by displacement, you will need to find a different liquid to estimate its density.[9] A solution or mixture will have different physical properties than each individual substance. So, by mixing water with other liquids or solids, a series of solutions with different densities can be prepared. Then, these can be used to examine which solids float and sink in each solution to estimate densities.

Typical densities of plastics and some useful solutions are given (Figure 8) so that you may identify the exact composition of your unknown once you estimate its density. For example,

Question: An unknown solid sinks in water, but floats in Solution 5 (13% w/w NaCl). Identify the solid.

Answer: It must have a density between that of water and Solution 5. Therefore, using Figure 8, the plastic must be polystyrene.

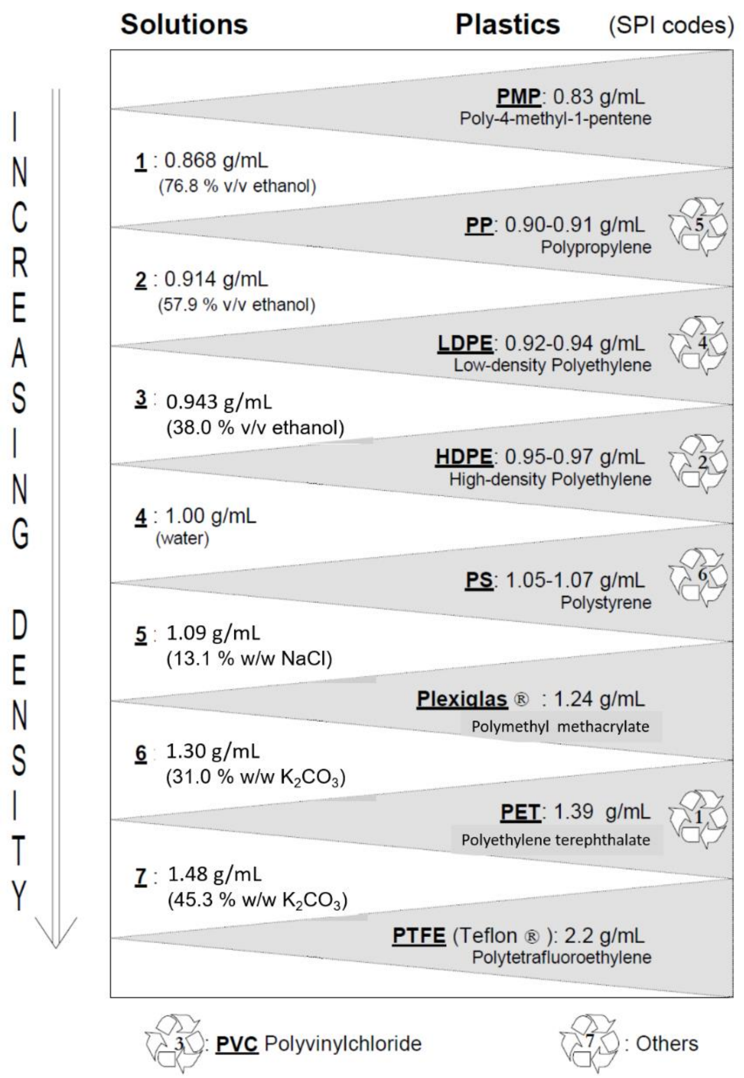


Figure 8. Densities of Plastics and Solutions for Comparison.
*% v/v and % w/w are measures of concentration. SPI Codes are now called Resin Identification Codes. Reprinted with permission from Hughes, E .A.; Ceretti, H. M.; Zalts, A. Floating Plastics: An Initial Chemistry Laboratory Experience. J. Chem. Ed., **2001**, 78 (4), 522. Copyright 2001 American Chemical Society.*[9]

The solution concentrations are given in % v/v, volume solute/volume solution percent or % w/w, weight solute/weight solution percent. For example:

$$76.8\ \%\ {}^{v}/_{v}\ ethanol = \frac{76.8\ mL\ of\ ethanol}{100.00\ mL\ of\ solution\ (ethanol + water)} \times 100\%$$

$$45.3\ \%\ {}^{w}/_{w}\ K_2CO_3 = \frac{33.110\ g\ K_2CO_3}{73.113\ g\ of\ solution\ (K_2CO_3 + water)} \times 100\%$$

You do not need to be able to perform these concentration calculations yet; just follow the procedures to mix the solutions with appropriate densities or use the solutions that will be ready in the laboratory.

You might like to start by observing whether your unknown plastics float in water. Then, decide which of the solutions (Table 2) to use in order to estimate density.

Table 2. Solutions for Density Comparison with Unknown Plastics.

Solution	**Composition**	**Expected Density at 20 °C (g/mL)**
1	76.8 % (v/v) Ethanol : water	0.868
2	57.9 % (v/v) Ethanol : water	0.914
3	38.0 % (v/v) Ethanol : water	0.943
4	Distilled water	1.00
5	13.1 % (w/w) sodium chloride (NaCl) : water	1.09
6	31.0 % (w/w) potassium carbonate (K_2CO_3) : water	1.30
7	45.3 % (w/w) potassium carbonate (K_2CO_3) : water	1.48

Experimental Procedure

Note: This experiment will take two weeks for completion. Parts A and B will be done the first week. Part C will be done the second week.

Wear goggles and gloves at all times.

Part A Basic Laboratory Techniques: Reading Common Glassware

Record the data from this section in Data Table 1.

1. At a station labeled 'Knowns', there should be two graduated cylinders and a buret, each filled with liquid. The measured volume for each instrument should be provided. Read each meniscus, record the reading including significant figures and units, and see if you agree with the provided value. If your results agree, then proceed to the next step, and if not, then consult your instructor.
2. At a station labeled 'Unknowns', there should be two graduated cylinders and a buret. Read the meniscus level for each of the instruments to the appropriate significant figures including units. Record the volumes.

Part B Determining the Density of Water at Room Temperature

List of Chemicals

- deionized (DI) water

List of Equipment and Glassware

- two 125-ml Erlenmeyer flasks
- two 100-ml beakers
- 10-ml volumetric pipet and bulb
- 25-ml graduated cylinder

Using a Volumetric Pipet

Record the data from this section in Data Table 2.

1. Obtain and label a clean, dry 125-mL Erlenmeyer flask as ‘Flask 1 water’ (marking tape and wax pencils/markers are located in a designated area in your lab).
2. Measure the mass of the flask on an analytical balance, and record the reading including units.
3. Fill a 100-mL beaker with deionized (DI) water. Using a clean, dry 10-mL volumetric pipet, measure 10.00 mL into the labeled flask following appropriate technique (Technique 5 Video Tech. 5 Solutions Using a Volumetric Pipet).
4. Record the mass of the flask with the water in (Technique 2 Using a Balance).
5. Repeat the process, measuring 10.00 mL DI water into Flask 1, and recording the mass each time until 50.00 mL of water total have been added to the flask.

Technique 2

Technique 5
Video Tech. 5

Using a Graduated Cylinder

Record the data from this section in Data Table 3.

1. Obtain and label a clean, dry 125-mL Erlenmeyer flask as ‘Flask 2 water’.
2. Measure the mass of the flask on an analytical balance, and record the reading.
3. Using a 25-mL graduated cylinder, measure 10.0 mL of DI water into Flask 2 (Technique 4 Using a Graduated Cylinder, Technique 3 Transferring Liquids). Record the mass of the flask with the water.
4. Repeat the process, measuring 10.0 mL DI water into the labeled flask, and recording the mass each time until 50.0 mL of water total have been added to the flask.

Technique 3

Technique 4

Recording the Temperature and Pressure to Determine Accepted Density

Record the data from this section in Data Table 4.

1. Near the instructor's desk or demonstration bench, there should be a thermometer suspended in water. Read and record the temperature of water with appropriate significant figures and units.
2. Also near the front of the room, locate the barometer and record the barometric pressure with appropriate significant figures and units.
3. Using Appendix 1, record the accepted density of water from the table at the closest temperature and pressure to your recorded values.

Part B Calculations

Your instructor may direct you to perform these calculations after the lab. Fill your final results into Data Table 5.

1. Fill in the cells in your data tables with your calculated values (the shaded cells in the tables). Remember to use the rules for calculating with significant figures. Show one example of each type of calculation below the tables.

2. Using the mass and volume data obtained for each flask, use Excel to plot mass versus volume following the directions above Data Table 5.

Clean up/Disposal

For cleanup, water may be disposed down the sink. Dry the outsides of the glassware. Return all glassware to its original location.

Part C Identifying Unknown Solids by Density

List of Chemicals

- deionized (DI) water
- two solutions of ethanol in water (76.8 %, 57.9 %)
- two solutions of potassium carbonate (K_2CO_3) in water (31.0 %, 45.3 %)
- 95% ethanol
- sodium chloride
- one set of five unknown plastics

List of Equipment and Glassware

- ruler
- 50-mL or 100-mL graduated cylinder
- 10-mL volumetric pipet with bulb
- Two 25-mL volumetric flasks
- eight 50-mL beakers with watch glasses
- beaker for waste with watch glass
- transfer pipet
- tweezers or forceps

Safety Precautions


Safety

- Wear safety goggles and gloves at all times.
- Ethanol is a flammable liquid. It is harmful by ingestion, inhalation, or skin absorption. It is an irritant to the eyes, nose, throat, and skin. Keep beakers covered with a watch glass (Figure 9).
- Potassium carbonate (K_2CO_3) solution is a strongly basic solution in water that can cause skin corrosion and eye damage.

Figure 9. Watch Glass and Placement on Beaker.

Record all the data for your unknowns (all the sections below) in Data Table 6.

Deciding on a Protocol

1. Your glassware must be clean and dry.
2. Examine the unknown objects, and write your observations of their appearance in your data table including their unknown number(s). Since you are analyzing a batch of plastics, it will help your instructor to know which object you are identifying with an accurate description of it.
3. Measure the mass of each one using the analytical balance, and record including all significant figures and the units.
4. Determine which protocol from the Scenario would be best for measuring each volume. Write the procedural steps that you used for each object and the resulting data in your data table.

Measuring with a Ruler

1. If you have a regular-sized object, align one edge of the object with the cm markings on the ruler. Set the end of the object at the zero marking. *Note: this procedure cannot be used with a small chip of plastic.*
2. Carefully, estimate the length, making sure to consider certain and uncertain digits. Record all significant figures and units.
3. Repeat for each side of the object.
4. Calculate the volume. Note that 1 mL = 1 cm^3.

Measuring Volume by Water Displacement

1. Choose a graduated cylinder with a height and width that can fit the unknown object and leave room for water to be measured. Add enough water to the graduated cylinder so that the unknown solid will be submerged when added. Measure and record the volume of the water carefully. *Note: this procedure cannot be used with a small chip of plastic nor can it be used with a piece too big for the cylinder.*
2. Then, tip the cylinder and slide the object into the water gently so that it does not crack the cylinder. Wait a few seconds for the water to run back down the sides of the cylinder; you might need to tap the side of the cylinder to encourage any residual drops to fall. Measure and record the volume of the water and object. The difference in volumes corresponds to the volume of the object.

Preparing Solutions for Density Comparison

Four solutions from Table 2 are pre-prepared for you in the lab. You will prepare the 38.0% ethanol and 13.1% sodium chloride solutions if you think they are necessary based upon your initial observations of your unknowns.

1. Collect two 25-mL volumetric flasks and label each with the name of the solution to be prepared.

2. *38.0 % ethanol solution (solution 3):*
    a. Obtain approximately 15 mL of 95% ethanol in a small beaker. Use it to condition a 10-mL volumetric pipet with 95% ethanol (Technique 5 Video Tech. 5 Using a Volumetric Pipet).
    b. Then, measure 10.00 mL into a 25-mL volumetric flask.
    c. Fill the volumetric flask to the mark with deionized (DI) water (Technique 6 Using a Volumetric Flask). Stopper and mix.
    d. Note that volumes for solutions are not always additive since two different liquids interact differently than the pure liquid alone, e.g., measuring 10.00 mL ethanol, you cannot expect to use 15.0 mL to fill the flask to 25.0 mL – it may take more.

Technique 5
Video Tech. 5

Technique 6

3. *13.1 % sodium chloride solution (solution 5)*:
    a. Weigh 3.6 g of sodium chloride (Technique 2 Using a Balance). Transfer it to a 25-mL volumetric flask.
    b. Add deionized water to the flask until it is approximately half-filled. Swirl the flask to dissolve the solid. Add more water gradually with stirring, but do not go past the mark.
    c. Once dissolved, fill the volumetric flask to the mark with deionized (DI) water (Technique 6 Using a Volumetric Flask). Stopper and mix.

4. Assume the densities of these solutions match those in Table 2.

Identification of plastics by Density Comparison

1. Collect seven watch glasses and seven 50-mL beakers, one for each of the solutions listed in Table 2. Label each beaker clearly with the solution name and density listed in Table 2.
2. Place approximately 25 mL of the corresponding solution in each beaker. Keep them well covered with a watch glass to prevent contamination.
3. Carefully submerge one of your unknown samples of plastic in the beaker containing distilled or deionized (DI) water (tweezers or forceps should be used); shake gently to dislodge any trapped air. Observe if the sample sinks or floats in this liquid, and record your observations.
4. Take out the sample. Dry the sample and the tweezers with a paper towel.
5. Decide which solution you should test it in next. You need to find two consecutive solutions: one in which it floats and one in which is sinks. Test it in as many solutions as you deem necessary. After testing in a solution, take out the sample with the tweezers, rinse it (and the tweezers) with water into a waste beaker, and dry with a paper towel.
6. Estimate each unknown density as intermediate between the last solution in which it sinks, and the first in which it floats. Use this value to identify your unknown sample (see Figure 8).

Clean up/Disposal

- Read the waste containers carefully to dispose the solutions in the correct container. Dispose the ethanol solutions in the Organic Waste container. Dispose the sodium chloride and potassium carbonate solutions in the Inorganic Waste container (Technique 11 Disposing Waste).
- Dispose the deionized water down the sink drain.
- Rinse and dry the five unknown plastics. Place them in their storage container and return the container to its original location.
- Rinse each piece of glassware with DI water, and collect the rinses in your waste beaker.
- Pour the contents of your waster beaker into the designated waste container(s).
- Wash all glassware with soap and water, and then rinse with deionized water (Technique 1 Cleaning Glassware). Dry the outsides of the glassware. Return all glassware to its place.

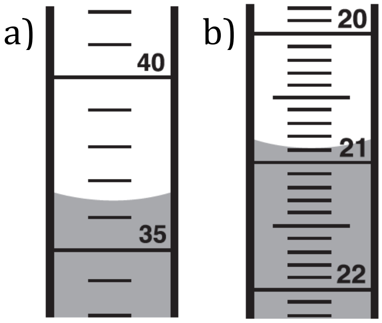
Technique 11

Technique 1

Pre-lab

The pre-lab assignment must be completed before you come to the lab.

1. Record the following volumes (in mL) to the appropriate significant figures.



2. Briefly explain how to use a volumetric pipet.
3. The mass of an Erlenmeyer flask is 85.135 g. After 10.00 mL of water is added to the flask, the mass of the flask and the water is 95.023 g. Calculate the density (in g/mL) of water.
4. Using the density calculated in question 3, calculate the percent error if the accepted density at this temperature is 0.9992 g/mL.
5. What does homogeneous mean? What do you think this means for the appearance of the solutions that you will use?
6. The mass of a volumetric flask is 40.638 g. After adding 15.0 mL of 95% ethanol and adding enough water to complete the volume to 50.00 mL, the flask weighed 88.219 g. What is the density of the mixture?
7. Print the data tables in the pages below and bring them to lab. Make them neat and legible so that you may communicate your results to others such as your instructor.

Post-lab

1. Instruments that were used to measure volume included a graduated cylinder, a volumetric pipet, and a buret, whereas a beaker does not provide volume with any reasonable certainty. Provide one explanation as to why this is true.
2. Among the two instruments that you used to measure density of water in Part B Determining the Density of Water at Room Temperature, which one was most accurate? What in the data supports your argument?
3. Which method, average or graph, was more accurate in determining the density of water?
4. Which has a greater effect on density of water: temperature or pressure? How do you know?
5. Briefly explain a process for how you would scale up your density technique to sort a whole batch of mixed plastics for recycling.
6. Conclusions: Once you have completed your experiment, you will need to prepare a report (see Appendix 6 How to Write a Lab Notebook). Your report should be less than 5 pages, be clear and readable to the instructor and anyone that would need your data, and include:
    - Title and lab partner names
    - Purpose
    - Any diagrams, figures, or tables with your lab data (units and significant figures are important, remember your Excel graph too)
    - Calculations (include one example calculation for each type)
    - Post-lab questions

    Conclusions (see Appendix 9 How to Write a Scientific Conclusion What concept were you investigating and how does it relate to the experimental procedure? How did you go about your work and why? This is **not** the details of your procedure repeated again, but discussion of the processes. For example, describe the methods for finding volume of the unknown objects. What is your conclusion for each part? Use the values you obtain as evidence in your reasoning. Statements like, "see data table for values" are not acceptable! Discuss the validity and reliability of your data in answering the question. Clearly state the identity of each unknown plastic.)

Experiment 1: Mass, Volume, and Density
Experimental Data and Calculations

Name: ______________________________ **Date:** ______________

Lab Partner: ______________________________ **Section:** ________

Print the data tables before lab. Remember to include units of measure with each entry, and to read and record each measurement to the full precision allowed by the instrument used. Make your work neat and legible so that you may communicate your results to others such as your instructor.

Data Table 1. Reading Common Glassware.

Known Readings	10 mL grad. cylinder	50 mL grad. cylinder	buret
Unknown Readings **Unk. #_____** **(if given)**	10 mL grad. cylinder	50 mL grad. cylinder	buret

Data Table 2. Measuring Volume with a Volumetric Pipet.

Mass of flask 1						
Mass of flask 1 + H_2O	▨					
Mass of H_2O	0.000 g					
Total Volume of H_2O	0.00 mL	10.00 mL	20.00 mL	30.00 mL	40.00 mL	50.00 mL
Density of H_2O	▨					
Mean of Density						
Standard Deviation						

(Note: the clear cells should contain your data; the shaded cells will contain calculated values.)

Data Table 3. Measuring Volume with a Graduated Cylinder.

Mass of flask 2						
Mass of flask 2 + H_2O	▨					
Mass of H_2O	0.000 g					
Total Volume H_2O (check the sig figs of your graduated cylinder)	0.0 mL	10.0 mL	20.0 mL	30.0 mL	40.0 mL	50.0 mL
Density of H_2O	▨					
Mean of Density						
Standard Deviation						

Show your work for one example of each type of calculation in Data Tables 2/3:

Data Table 4. Temperature and Pressure Measurements.

Barometric Pressure	
Water Temperature	
Density from Appendix 1	

- Using the mass and volume data obtained for each flask (from
- Data Table 2 and Data Table 3), use Excel to plot two mass (y-axis) versus volume (x-axis) scatter plots (see Appendix 7 Using Excel).
    - Add a trend line for each plot; the slope of each line will be the density. Make sure to include the line equation and the coefficient of determination (R-squared value).
    - You can place both lines on the same plot. Make sure to use a legend to label each line.
    - Add a title to the plot.
    - Print the Excel plot, and include it in your lab report.
  - Use the slope of each line to fill in the density in Data Table 5. Use the accepted value for density from Appendix 1 (Data Table 4) to calculate the relative percent error.

Data Table 5. Relative Percent Error for the Density of Water.

	Density from Excel Plot	**Rel. % Error**
Flask 1 (using pipet)		
Flask 2 (using graduated cylinder)		

Show your work for one example of each calculation:

Data Table 6. Density and Identity of Unknown Plastics in Set Number ___________.

Unknowns	Observations	Mass	Volume	Density	Identity
example object	clear, colorless flake	0.2790 g	too small to measure by ruler or displacement	• floats on water • sinks in sol'n 3 • density must be between 0.945 and 1.00 g/mL	HDPE
Object 1					
Object 2					
Object 3					
Object 4					
Object 5					

Show your work for one example of each type of calculation:

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Experiment 2:
Empirical Formulas

Version 3a

Diego J. Díaz López, Ph.D., Laura B. Sessions, Ph.D., and Eileen Pérez, Ph.D.

Two of the basic tenets of atomic theory: Lavoisier's Law of Conservation of Mass and Proust's Law of Definite Proportions are applied to chemical reactions involving magnesium and a copper salt. The data obtained from the reactions can be used to determine the empirical formulas of the compounds and the amount of water in a hydrate.

Objectives

- To determine the empirical formulas of compounds
- To determine the percent composition of a reaction product

Learning Outcomes

- To apply the Law of Conservation of Mass to a chemical reaction.
- To apply the Law of Definite Proportions to determine the empirical formulas of compounds.
- To determine the empirical formula of a compound.
- To learn how to determine the empirical formula of a hydrate.

Definitions

- **decanting** – gradually pouring off the supernatant, leaving the solid in the original container
- **empirical formula** – chemical formula based on the lowest possible integer coefficient of elements
- **formula unit** - the ratio of atoms in an ionic compound, the chemical formula; although it does not represent the whole crystal structure of an ionic compound, it is useful for stoichiometric calculations
- **hydrate** – inorganic compound containing a determined ratio of water molecules per formula unit
- **Law of conservation of mass** – establishes that mass is preserved during the course of a chemical reaction; that is, mass is not created nor destroyed in chemical reactions
- **Law of definite proportions** – establishes that chemical compounds always have the same mass ratio of elements
- **molecular formula** – chemical formula based on the actual ratio of its elements
- **oxidation** – loss of electrons in an oxidation-reduction (redox) reaction
- **oxidizing agent** – element that causes another element to be oxidized by being reduced (accepting the other element's electron or electrons)
- **reduction** – gain of electrons in an oxidation-reduction (redox) reaction
- **reducing agent** – element that causes another element to be reduced by being oxidized (giving away its electron or electrons to the other element)
- **supernatant** – liquid lying over the solid in a chemical reaction or process

Introduction

Many metal ions exhibit bright, beautiful colors when excited by a hot flame. This excitation is the basic principle of pyrotechnic colorants or fireworks. In fireworks, the cation present determines the color shown, while the anion often can alter the temperature of the flame, helping to adjust the brightness and duration of the firework. These materials are compacted into pods called stars, consisting of a blend of oxidizing agent, reducing agent, coloring agent (metal salt), and binders. When ignited, the stars produce both sound and light effects. The appearance of a firework is determined by its stars. Blue color is often produced from the salts of the copper(I) ion, especially the chloride. Some other metals such as titanium and magnesium are often used to improve the brightness of the fireworks, or to produce bright whites. Although magnesium produces beautiful bright whites, its natural oxidation or common alloying can affect its brightness. The Light Me Up Fireworks company had to recall a batch of their fireworks as the white stars did not produce the desired results. This batch caused a major embarrassment in many Fourth of July celebrations as the white color appeared rather bleak, and the blues appeared more greenish than blue. As a chemist for the Light Me Up Fireworks Company, you were asked to assess the failure. Your colleagues believe that the materials were accidentally swapped and that different materials were used. You strongly believe that the cause of the problem was contamination with an oxidizing agent that caused the undesired oxidation of the materials used. Burning of the magnesium should generate the oxide in an oxidation-reduction reaction; and if part of the material was already oxidized, it will not produce a bright white flame. After all, magnesium can oxidize naturally to the oxide by air and in the presence of many oxidizing agents. The undesired oxidation of the copper ion in your salt can give colors that range from green to purplish. You will follow an experiment to test the formula of the materials used in the fireworks.

Chemists write formulas in different ways. The most common is the molecular formula. The molecular formula shows the actual number of atoms of each element present in the formula. A simple form of a chemical formula is an empirical formula. An empirical formula consists of the lowest possible integer ratio of the elements forming the compound. The empirical formula is not how a compound exists, and does not represent the actual structure of the compound, but it is useful since it can be determined easily by experiment. For example, the chemical compound benzene has a molecular formula of C_6H_6. The lowest possible integer ratio between the carbon and the hydrogen will be 1:1, thus benzene has an empirical formula of CH.

Two of the basic foundations of our atomic theory are Antoine Lavoisier's law of conservation of mass, and Joseph Proust's law of definite proportions (also known as law of constant composition or law of definite composition). The law of conservation of mass states that matter is not created nor destroyed, but preserved during the course of a chemical reaction. The law of definite proportions states that a chemical compound will always have the same ratios of its elements. We can observe that benzene will always consist of 92.2 % carbon by mass, and 7.8 % hydrogen by mass.

Each C_6H_6 molecule will contain six carbon atoms, and six hydrogen atoms. Using the molar masses from the periodic table, we obtain:

$$mass\ carbon = 6 \times 12.01\ g/mol = 72.06\ g/mol \text{ , and}$$

$$mass\ hydrogen = 6 \times 1.01\ g/mol = 6.06\ g/mol \text{ , thus}$$

$$\%\,C = \frac{72.06\ g\ C}{72.06\ g\ C + 6.06\ g\ H} \times 100 = 92.24\% \qquad \%\,H = \frac{6.06\ g\ H}{72.06\ g\ C + 6.06\ g\ H} \times 100 = 7.76\%$$

No matter how large or small the sample is, benzene will always have this ratio or percent. The law of definite proportions and the law of conservation of mass enable us to determine the formula of a compound if we are able to determine the masses, or the percentages of the different elements in the formula. Once we obtain the masses for the elements, we can use the elements' molar masses to determine the number of moles of each element and determine the ratio between them.

For example, a chemist found that a given compound is composed of 0.410 g Al and 1.590 g Cl. What is the empirical formula?

1. We begin by determining the number of moles of each element.
2. $n_{Al} = 0.410\ g \left(\frac{1\ mol}{26.98\ g}\right) = 0.0152\ mol \qquad n_{Cl} = 1.590\ g \left(\frac{1\ mol}{35.45\ g}\right) = 0.04485\ mol$
3. This determines a ratio of 0.0152 mol Al: 0.04485 mol Cl. We want to turn this ratio into integers. We accomplish that by dividing both numbers by the smallest one.
4. $Al_{\frac{0.0152}{0.0152}}Cl_{\frac{0.04485}{0.0152}} \quad or \quad AlCl_3$
5. We should expect that due to experimental errors, the ratios will not be exact integer numbers, but within the uncertainty, they can be rounded to the closest integers. If the ratio of any of the elements is close to 0.5 (1.5, for example), the ratios of all the elements in the compound need to be multiplied by a factor of 2 to turn them into integer ratios.

Let's consider another example. In this experiment, in addition to the law of definite proportion, we will also need to apply the law of conservation of mass to find the chemical formula of a hydrate. Hydrates are salts with a strong affinity towards water. They have a fixed ratio of water molecules. For example, $CaCl_2{\cdot}2H_2O$ (calcium chloride dihydrate) has two water molecules per formula unit. In the case of hydrates, the water of hydration will not be removed by air drying. However, these water molecules can be removed under more intense heating. The masses of the compound before and after heating to remove the water molecules allows us to obtain the mass of salt, and the mass of water. We can determine the number of moles of salt, and the number of moles of water in order to find the ratio of water molecules per formula unit.

In today's experiment, you will apply these concepts in order to determine the formulas of the products obtained by burning magnesium and dehydrating a copper salt.

Experimental Procedure

Part A Empirical formula of the product of the reaction of Mg with O_2

Record the data of Part A in Table 1.

Safety Precautions

Allow time for glassware and equipment to cool

Techniques

- Technique 2: Using a balance
- Technique 9: Using a Bunsen burner

Technique

List of Chemicals

- deionized (DI) water
- magnesium ribbon

Equipment

- two crucibles and lids
- Bunsen burner
- striker
- clay triangle
- ring support
- stand
- hot pad
- analytical balance
- disposable transfer pipet
- crucible tongs

1. Familiarize yourself with the use of the Bunsen burner. Following instructions of Technique 9 Using the Bunsen Burner, turn on and adjust height of flame and air so all three cones visible. Show flame to instructor for approval/signature on Experimental Data and Calculations sheet.

Technique 9

2. Obtain a clean crucible and lid. Check the crucible for cracks. A cracked or chipped crucible will break upon heating or cooling. Support the crucible and lid on a clay triangle and heat with an intense flame for 5 minutes (see Figure 1). NOTE: Start with low heat for about 3 minutes and gently increase to intense for another 2 minutes. Make sure that the lid is slightly opened. This "firing" process will remove any contaminants from the crucible that would affect mass change during the experiment.

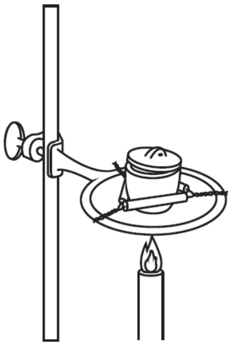


Figure 1. Set-up for heating. The burner is positioned several inches from the crucible.

3. Allow the crucible to cool on the clay triangle for 2-3 minutes. Then, place hot crucibles and hot lids on hot pads - never directly on the bench top. Allow them to cool to room temperature.
4. Measure the mass of the fired, cool crucible and lid. Use only clean, dry crucible tongs to handle the crucible and lid for the remainder of the experiment.
5. Cut a 10 to 12 cm strip of Mg. Polish it with steel wool or sand paper.

Technique 2

6. Curl the Mg as tightly as possible. Make sure that the coil lies flat on the bottom of the crucible. Measure and record the mass of the Mg, crucible, and lid.
7. Heat over the flame for complete reaction. You will need to slightly lift the lid to allow oxygen into the reaction once per minute. Make sure that the lid is not fully removed as Mg burns very bright and may cause temporary blindness.
8. When the reaction appears complete (no visible change and sample looks like ash), allow the crucible to cool. Weigh the burned product, crucible, and lid.
9. Make sure that the sample has cooled, and add a couple of drops of DI water. There should not be standing water in the crucible. Burn again for 5 minutes without the lid. Cool the product after the second burn and weigh the product with the crucible and lid for a second time. If the difference in mass between the first and second burn is less than ±0.010 g, you may continue and record the second reading as the mass of the product. If the difference is larger, do a third burn and record the mass as the mass of the product.
10. Repeat the experiment with a second piece of Mg.

Part B Determination of the Amount of Water in a $CuCl_2 \cdot nH_2O$ Compound

Record the data of this section in Table 2.

Safety Precautions

Allow time for glassware and equipment to cool

Techniques

- Technique 2: Using a balance

Technique 2

List of Chemicals

- deionized (DI) water
- copper (II) chloride hydrate crystals

Equipment

- hot plate
- two evaporating dishes
- hot pad
- analytical balance
- crucible tongs or hot hand protector

1. Weigh a clean evaporating dish. Add approximately 1 g of $CuCl_2 \cdot nH_2O$ to the evaporating dish and record the mass to the full precision of the balance.
2. Place the evaporating dish on a hot plate. Heat for 10 minutes at about 2/3 of the maximum power (300 °C setting). Note: The color of the solid will gradually change from green to brown.
3. When only a small amount of the solid remains green, stir the mixture with a glass stirring rod to move the unreacted parts on top toward the bottom of the evaporating dish for better heating. Note: avoid stirring the crystals until most are brown since the hydrate tends to stick to the rod.
4. Using tongs or a hand protector remove the evaporating dish from the heat and place it in a desiccator. Let it cool for five minutes.
5. After five minutes, remove the evaporating dish from the desiccator and weigh the evaporating dish.

6. Place the evaporating dish in the hot plate and heat again for five minutes at 2/3 of maximum power (300 °C).
7. Using tongs or a hand protector place the evaporating dish in the desiccator for an additional five minutes.
8. Remove the evaporating dish from the desiccator and re-weigh it.
9. If the mass difference between the two measurements is larger than 0.01 g, re-heat a third time for an additional 3 minutes. Allow it to cool in the desiccator. Re-weigh.
10. Repeat above procedure one more time with a new sample. (If both evaporating dishes fit in the desiccator, heat both trials at the same time.)
11. Determine the mass of the dehydrated samples.
12. Repeat the experiment with a second sample of $CuCl_2 \cdot nH_2O$.

Clean up/Disposal

- Dispose of all materials on the corresponding waste containers as indicated by your instructor.
- Wash all glassware with soap and water, and then rinse with deionized water (Technique 1 Cleaning Glassware). Dry the outsides of the glassware. Return all glassware to its place.

Pre-lab

1. When using a crucible and lid, why must the crucible and lid be inspected for cracks before using?
2. What is meant by "firing the crucible"? Why must we "fire" the crucible before using it?
3. A 1.30 g sample of titanium chemically combines with chlorine gas to form 5.16 g of titanium chloride. (a) What is the empirical formula of titanium chloride? (b) What is the percent by mass of titanium and the percent by mass of chloride in the sample?
4. A 0.500 g sample of tin foil reacted with oxygen to give 0.635 g of product. (a) What is the empirical formula of the tin oxide? (b) What is the percent by mass of tin and the percent by mass of oxygen in the sample?
5. Epsom salt is commonly purchased in the pharmacy for a variety of uses, anti-inflammatory, laxative, and cosmetic. Epsom salt is a hydrated salt of magnesium sulfate. If 2.000 g of Epsom salt are heated to remove the waters of hydration, 0.977 g of the anhydrous (without water) magnesium sulfate, $MgSO_4$, were obtained. Calculate the number of waters of hydration, and write the chemical formula of the Epsom salt.

Post-lab

In the data/calculations section:

Include the data collected in the lab and complete the calculations.
Write a conclusion (see Appendix 9 Writing a Laboratory Conclusion).
After determination of the empirical formulas, was the charge of the copper and magnesium what was expected? Do the results support the predicted charge?
Answer the following Post-Lab Questions.

1. In part A, the crucible was not "fired" before burning the magnesium. When the magnesium was burned, volatile impurities in the crucible were burned off. Will this error increase, decrease, or not affect the ratio of magnesium to oxygen determined and the empirical formula found?
2. In part A, a student did not completely burn all the magnesium and some un-burned magnesium remained. How will this affect the empirical formula found?
3. In part A, a student forgot to allow some air (oxygen) to get into the crucible while burning the magnesium. The oxygen insufficiency resulted in the formation of the magnesium nitride (Mg_3N_2) instead of the magnesium oxide. How will this error affect the magnesium-to-oxygen ratio and the empirical formula found?
4. In part B, a student burned the hydrate over a very intense heat, and some of the copper(II) chloride was turned into a copper(II) oxide. Will the number of waters of hydration determined be too high, too low, or remain the same?
5. In part B, a student forgot to place the heated sample in the dessicator while cooling, and some water was re-absorbed by the copper (II) chloride. Will the number of waters of hydration determined be too high, too low, or remain the same?

Experiment 2:
Empirical Formulas
Experimental Data and Calculations

Version 3a

Name: ______________________ **Date:** ______________

Lab Partner: ______________________ **Section:** ________

Experimental Data and Calculations

Proper use of the Bunsen Burner - Professor's signature: ________________

Table 1. Empirical formula of the Product of the Reaction of Mg with O_2

	Trial 1*	Trial 2
mass of lid		
mass of crucible + lid		
mass of crucible + lid + Mg		
mass of Mg		
moles of Mg		
mass of burned Mg product + lid + crucible		
1st mass recording after heating		
2nd mass recording after heating		
3rd mass recording after heating (if needed)		
mass of oxygen		
moles of oxygen		
formula of the magnesium oxide		

Name: ____

*Show calculations for Table 1 trial 1.

Table 2. Empirical Formula of a Copper Hydrate

	Trial 1*	Trial 2
mass of evaporating dish		
mass of evaporating dish + $CuCl_2{\cdot}nH_2O$		
mass of copper(II) chloride hydrate		
mass of copper(II) chloride + evaporating dish (after heating) (g)		
1st mass recording after heating		
2nd mass recording after heating		
3rd mass recording after heating (if needed)		
mass of H_2O		
moles of H_2O		
mass of anhydrous $CuCl_2$		
moles of $CuCl_2$		
formula of the copper(II) chloride hydrate		

Name: ____

*Show calculations for Table 2 trial 1.

VALENCIA COLLEGE

Experiment 3:
Electrolytes and Nonelectrolytes

Version 4a

Eileen Pérez, Ph.D.

In this experiment, you will study the conductivity of solutions of strong electrolytes, weak electrolytes, and non-electrolytes. You will also measure the pH and perform some tests that will allow you to observe chemical reactivity. To have a baseline or control, you will perform the same tests on deionized water. Then, you will study an unknown and compare results to identify it.

Objectives

- Discover some properties of strong electrolytes, weak electrolytes, and non-electrolytes by observing their behaviors in solutions.
- Write equations for the ionization of strong and weak electrolytes in water.
- Write equations for the dissolution of nonelectrolytes in water.
- Learn how to measure conductivity and pH.
- Compare concentration of hydronium ion produced by strong and weak acids.
- Observe precipitation reactions.
- Write the chemical formula of products formed from double replacement reactions.
- Determine identity of an unknown.

Learning Outcomes

- Understand the nature of units of measurement and apply rules for significant figures.
- Understand the nature of matter and its underlying physical and chemical characteristics.
- Understand the nature and characteristics of chemical bonds.
- Understand/apply the rules to write chemical formulas of ionic compounds.
- Employ conceptual learning outcomes and perform essential lab techniques in laboratory setting.

Definitions

- **Acids** – molecular compounds that are able to donate a hydrogen ion, H^+, when dissolved in water, thereby increasing the amount of free H^+ in the solution. They are written the hydrogen first to denote that the substance is an acid.
- **Aqueous solution** - a solution in which water the is the solvent
- **Conductivity** – the ability of a material to allow flow of electricity, heat, sound, ions, etc.; this experiment focuses on flow of electricity
- **Control** – the standard for comparison in an experiment; treated in the same way the experiment is performed but without the variable that is being tested
- **Dissociation** – the process of a solute mixing and dissociating (breaking apart into ions) within a solvent to form a solution
- **Dissolution** – the process of a solute mixing and dispersing within a solvent to form a solution
- **Electrolytes** – substances that conduct electricity when dissolved in water

- **Formula unit** – the smallest, electrically neutral collection of ions in an ionic compound
- **Ionic substance** – compound composed of cations and anions chemically bonded through electrostatic attraction
- **Ions** – atoms or a group of bonded atoms with a net charge
- **Ionization** – process of gaining or losing electrons to become an ion
- **Molarity, M** – unit of concentration; expressed as moles of solute per liters of solution, mol/L
- **Molecular substance** (also known as covalent substance) – compound composed of two or more nonmetals sharing electrons (covalently bonded)
- **Molecules** – smallest particle of a molecular compound that maintains the characteristics of the molecular compound (also applies to multi-atomic elements such as N_2 or S_8)
- **Nonelectrolytes** – substances that do not conduct electricity when dissolved in water because they do not ionize; molecular substances that are not acids or bases are nonelectrolytes
- **pH** – measurement of the concentration of hydrogen ions, H^+, in solution. The pH scale generally ranges from 0 to 14 (although concentrated acids and bases have values below and above this range). Neutral solutions have a pH of 7. A pH value below 7 means that the solution is acidic; the smaller the value the more acidic. A pH value above 7 means that the solution is basic; the larger the value the more basic.
    - $pH = -log(M_{H^+})$, where M = molarity
- **Precipitate** – solid, insoluble compound that forms in a solution and settles on the bottom of the container
- **Reagent** – chemical substance used to detect or measure the presence of another substance by causing a reaction
- **Solute** – the component in lesser amount in a solution
- **Solution** – a homogeneous mixture of solute(s) and solvent
- **Solvent** – the major component of a solution
- **Strong electrolytes** – substances that dissolve in water completely or almost completely, forming ions. They are good conductors of electricity. Ionic compounds (including strong bases) and strong acids are strong electrolytes.
- **Weak electrolytes** – substances that dissolve in water, but do not completely ionize and therefore only weakly conduct electricity in solution

Introduction

You work as a Quality Control (QC) Chemist for EverBrite, a manufacturer of over-the-counter products, such as EverBrite Toothpaste, EverBrite Insta-Denture Grip, and EverBrite Scalp Refreshing Anti-Dandruff Shampoo and Conditioner. As QC chemist, your day-to-day responsibilities include performing tests on raw materials, bulk formulations, finished products, stability samples, and any other samples from EverBrite's plant operations that require chemical analysis. You report to the QC Laboratory Supervisor.

The QC Lab Supervisor is upset because an old 55-gallon drum was found in the back of the warehouse with a partially torn label. The drum is about half-filled with a clear solution. While this is a serious violation of EverBrite's Storage and Traceability SOPs (Standard Operating Procedures), the problem is exacerbated because the FDA (Food and Drug Administration) is scheduled to visit the facility by the end of the week. The QC supervisor has given your QC team the responsibility of determining the identity of the solution so that it can be labeled and disposed of properly.

Fortunately, the receiving date on the label was still legible as well as the supplier's logo. After reviewing the Receiving Logs for that day, your team was able to narrow down the possible identity of the unknown solution to six solutions: 0.50 M glucose ($C_6H_{12}O_6$), 50 % isopropyl alcohol (C_3H_8O), 0.50 M acetic acid ($HC_2H_3O_2$), 0.50 M hydrochloric acid (HCl), 0.50 M magnesium sulfate ($MgSO_4$), and 0.50 M aluminum chloride ($AlCl_3$).

Your team will explore the characteristics of these eight solutions, referred to as known solutions from now on, and compare those characteristics with the unknown to determine its identity.

Electrolytes and Nonelectrolytes:

A solution is formed when a solute is dissolved in a solvent. The solute appears to disappear in the solvent, but if you watch closely, as more solute is added, the volume of the solution often increases. As the solute interacts with the solvent, the attractive forces within the solute (solute-solute attractions) compete with the attractive forces between the solute and the solvent. If this latter attraction (solute-solvent attraction) is stronger than the former, the solute particles disperse throughout the solvent. When this happens, we simply state that the solute dissolved.

Consider, for example, a molecular substance such as sucrose, $C_{12}H_{22}O_{11}$, commonly referred to as table sugar. From experience you know that table sugar is water-soluble, that is, it dissolves in water. Let's consider what happens to one teaspoon (4.2 grams) of sucrose, at the particulate level, when it dissolves in 1L of water. This sucrose sample contains 7.4 x 10^{21} molecules of sucrose:

$$?\,molecules\ C_{12}H_{22}O_{11} = 4.2\ g\ \times \frac{1\ mol}{342.296\ g} \times \frac{6.022 \times 10^{23} molecules}{1\ mol}$$

$$= 7.4 \times 10^{21}\ molecules\ C_{12}H_{22}O_{11}$$

As the solid dissolves, the sucrose molecules separate from one another and disperse throughout the water, but each sucrose molecule stays intact; each molecule continues to be chemically bonded within, and each has the chemical formula of $C_{12}H_{22}O_{11}$. We can represent this process with the following chemical equation:

$$C_{12}H_{22}O_{11}\ (s)\ \overset{H_2O}{\rightarrow}\ C_{12}H_{22}O_{11}(aq)$$

Where (s) means solid, and (aq) means aqueous solution.

Now consider what happens to an ionic substance such as sodium chloride, NaCl, commonly referred to as table salt. From experience, you also know that table salt is water-soluble. One teaspoon (5.7g) of sodium chloride contains 5.9 x 10^{22} formula units of NaCl:

$$?\,formula\ units\ NaCl = 5.7\ g\ \times \frac{1\ mol}{58.44\ g} \times \frac{6.022 \times 10^{23} formula\ units}{1\ mol}$$

$$= 5.9 \times 10^{22}\ formula\ units\ NaCl$$

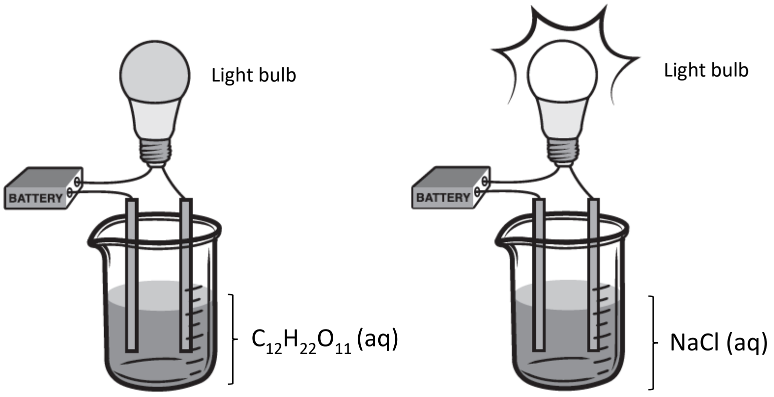
Like sugar, as the solid salt dissolves, its formula units separate from one another; unlike sugar though, the formula units break apart freeing up the individual ions:

$$NaCl\ (s)\ \overset{H_2O}{\rightarrow}\ Na^{+}(aq) +\ Cl^{-}(aq)$$

So now there are actually 1.2×10^{23} free ions dispersed in the water:

$$total\ ions\ = 5.9 \times 10^{22}\ ions\ of\ Na^{+} +\ 5.9 \times 10^{22}\ ions\ of\ Cl^{-}\ = 11.8 \times 10^{22}\ ions$$
$$= 1.2 \times 10^{23}\ ions$$

Solutions of molecular substances, such as $C_{12}H_{22}O_{11}$, do not conduct electricity (see Figure 1a) therefore they are called nonelectrolytes, whereas solutions of ionic substances, such as NaCl, do conduct electricity (see Figure 1b). This type of substance, called electrolytes, conduct electricity because the dissolved ions act as charge carriers, allowing the solution to conduct electricity.[1]



1a. Does not conduct electricity *1b. Conducts electricity*

***Figure 1**. Behavior of two solutions in an open circuit*

Strong Electrolytes and Weak Electrolytes:

A strong electrolyte is a substance that dissociates or ionizes to a very large extent when dissolved in water. Sodium chloride is an example of a strong electrolyte.

A weak electrolyte is a substance that dissociates or ionizes to a relatively low percentage (<<100% ionization) when dissolved in water. Hydrofluoric acid, HF, is an example of a weak electrolyte. This process is represented in the following equation:

$$\text{HF (aq)} \rightleftharpoons H^{+}(aq) + F^{-}(aq)$$

Notice the difference in the arrows used between this reaction and the NaCl reaction above: we use equilibrium arrows (⇌) to differentiate a weak electrolyte from a strong electrolyte. Table 1 below states which substances are electrolytes and which are nonelectrolytes.

Table 1. Classification of Substances as Electrolytes and Nonelectrolytes

Nonelectrolytes	• Molecular substances that are not acids or bases
Electrolytes	• Ionic compounds • Molecular compounds that are acids and bases
• **Strong electrolytes**	• Ionic compounds • Strong acids • Strong bases
• **Weak electrolytes**	• Weak acids • Weak bases

Note: Appendix 4 lists the most common strong acids and strong bases.

Notice that some acids are strong electrolytes while others are weak electrolytes. The percent ionization of an acid can help us determine if an acid is a strong acid or a weak acid. An acid with a high percent ionization is a strong acid. Since pH paper will be used in this experiment, an acid with a percent ionization $\geq$50% should be classified as a strong acid.

The percent ionization can be calculated as follows:

$$\%\ dissociation_{HA} = \frac{\text{concentration of hydrogen ion}}{\text{initial concentration of the acid}} \times 100 = \frac{M_{H^+}}{M_{HA}} \times 100$$

The pH value allows us to determine the concentration of hydrogen ion:

$$pH = -log(M_{H^+})$$

Example 1: Using pH paper, a 0.50 M solution of citric acid, $H_3C_6H_5O_7$, has a pH of 4.

a) Determine the concentration of hydrogen ion.
b) Determine the percent of ionization of this solution.
c) Classify citric acid as a strong or a weak acid.

Answers:

a) To find the concentration of hydrogen ion we use the pH equation:

$$pH = -log(M_{H^+})$$

Rearrange to solve for concentration of hydrogen ion, and substitute the pH value:

$$M_{H^+} = 10^{-pH}$$

$$M_{H^+} = 10^{-4} = 1 \times 10^{-4} M$$

b) $\%\ dissociation_{HA} = \frac{M_{H^+}}{M_{HA}} \times 100 = \frac{1 \times 10^{-4} M}{0.50\ M} \times 100 = 0.02\ \%$

c) Citric acid is weak acid.

Conductivity Test[2]:

When the conductivity probe is placed in a solution that contains ions, and thus can conduct electricity, an electrical circuit is completed across the electrodes that are located on either side of the hole near the bottom of the sensor body. This results in a conductivity value that can be read by the detector. The unit of conductivity used in this experiment is microsiemens per centimeter, µS/cm. The size of the conductivity value depends on the ability of the aqueous solution to conduct electricity. Strong electrolytes produce large numbers of ions, which result in high conductivity values. Weak electrolytes have a low conductivity because they only partially dissociate or ionize producing a lower number of ions. Non-electrolytes have no conductivity.

pH Test:

The pH will be measured using pH paper.

Chemical Tests:

You will perform several tests on each known solution to determine if they react or not with the test reagent. Some **evidence that a chemical reaction occurs** include:

- Gas evolution. Bubbling or "fizzing" is observed. The gas quickly leaves the solution, so observe carefully when adding the test reagent. Sometimes the gas has a distinctive odor.
- Heat exchange. If the container feels warmer to the touch after the reaction, the reaction released heat (exothermic); if the container feels cooler to the touch, the reaction absorbed heat (endothermic).
- Color change. Sometimes a permanent color change is evidence of a reaction. Do not confuse this with a physical change such as dilution. (For example, a little more water added to a blue solution will make it look lighter because it was diluted, which is a physical change; a completely different color or a change in hue – sky blue to royal blue, for example – might mean that a chemical reaction occurred.)
- A change in pH of at least 2 or more pH units usually indicates a chemical change.
- Formation (or disappearance) of a precipitate. (Sometimes small solid particles stay suspended in the solution rather than precipitating to the bottom. We refer to this as cloudy.) If you cannot see through the solution, a solid (precipitate or cloudy) has been formed. Appendix 2 in conjunction with Appendix 3 can help you determine the chemical formula of the solid formed.

Since the unknown solution is one of the 6 solutions that will be investigated through chemical tests, comparison of results obtained from the chemical tests, along with pH and conductivity values, will allow for proper identification of the unknown solution.

Techniques

- Technique 1: Cleaning glassware
- Technique 11: Disposing chemical waste
- Technique 12: Using pH paper
- Technique 19: Using the LabQuest data collector with conductivity probe

List of Chemicals

- 0.50 M glucose ($C_6H_{12}O_6$)
- 50 % isopropyl alcohol (C_3H_8O)
- 0.50 M acetic acid ($HC_2H_3O_2$)
- 0.50 M hydrochloric acid (HCl)
- 0.50 M magnesium sulfate ($MgSO_4$)
- 0.50 M aluminum chloride ($AlCl_3$)
- 0.2 M silver nitrate ($AgNO_3$)
- 0.2 M sodium carbonate (Na_2CO_3)
- 3 M ammonium hydroxide (NH_4OH)
- 1,000 µS NaCl standard solution
- Benedict's Reagent
- chromic acid (keep in hood)

List of Equipment and Glassware

- seven 18 mm x 150 mm test tubes
- two 13 mm x 100 mm test tubes
- one test-tube rack
- one beaker for Waste

- one 250-mL beaker
- LabQuest Data Collector
- one conductivity probe
- instructions sheet for LabQuest Data Collector with conductivity probe
- pH paper
- one stirring rod
- one thermometer
- one test tube holder
- four disposable pipets
- one 24-well plate
- one watch glass

Experimental Procedure

Part A: Collecting the Known Solutions and DI Water

1. Obtain seven 18 x 150 mm test tubes, a test-tube rack, and one beaker to use as waste container.
2. Clean and rinse the test tubes with deionized (DI) water. Shake out excess water and dry the outside of the test tubes (a few drops of water left inside will have a negligible effect in this experiment).

   Label them as follows:
    1. $C_6H_{12}O_6$
    2. C_3H_8O
    3. $HC_2H_3O_2$
    4. HCl
    5. $MgSO_4$
    6. $AlCl_3$
    7. H_2O

GOLDEN LAB RULE:
Be frugal: take only as much as needed. Solutions must never be returned to stock bottles.

3. Half-fill each test tube with the corresponding solution, all of which are 0.50 M concentration, except for isopropyl alcohol which is 50 %. Add deionized water in test tube 7. Since the solvent for known solutions 1-6 is deionized (DI) water, it will be useful to measure the conductivity and pH of deionized water. Results different from DI water will then clearly belong to the compound dissolved in it.

Part B: Determination of the Conductivity of the Known Solutions

1. Record the conductivity of the six known solutions and deionized water in Experimental Data Table 1.
2. Obtain a LabQuest Data Collector, conductivity probe, a 1,000 µS NaCl standard solution, and instructions sheet.
3. Set up and calibrate the LabQuest Data Collector with the conductivity probe following the instructions.

Technique 19

4. Carefully hold the conductivity probe in the test tube containing DI water (#7). Make sure that the opening near the bottom of the probe is submerged in the DI water. Be careful that the test tube does not overflow. Swirl the probe to remove any trapped air bubbles.
5. Record the conductivity, in µS/cm.
6. Remove probe, rinse into the waste beaker, and dry.

GOLDEN LAB RULE:
Prevent messes: hold the probe above the test tube so that drops fall into the test tube.

7. Measure the conductivity of the 6 known solutions following steps B4 – B6.
8. Leave the conductivity probe and LabQuest Data Collector connected. You will probably use it again in Part E.

   Note 1: *The readings will normally fluctuate quite a bit. Typically, wait about 30 seconds and then record the reading. Try to read the conductivity to the nearest 10, but for conductivities greater than 1000 you may not be able to do any better than to record it to the nearest 100.*

   Note 2: *Acids have unusually high conductivity values. Reasons go beyond the scope of this experiment. If you are curious, explanations are offered by Jeffery et al.*[3] *and Atkins.*[4]

Part C: Determination of the pH of the Known Solutions

1. Record the pH of the six known solutions and deionized water in Experimental Data Table 1.
2. Obtain pH paper and a clean dry stirring rod.
3. Remove a strip of pH paper about 3 inches long. Place the strip on a clean dry watch glass or a paper towel.
4. Dip the stirring rod into the test tube containing DI water (#7); then touch the pH paper with it (near the left edge).

Technique 12

5. Immediately compare the color on the center of the wet spot with the key on the casing of the pH paper. If the color looks like it is between two adjacent colors, estimate it to the first decimal place (for example, if it looks like it is between 3 and 4, estimate it as 3.5).
6. Record the pH.
7. Using your wash bottle, rinse the stirring rod over waste beaker and dry it with a paper towel.
8. Repeat steps C4 to C7 until you have measured the pH of the 6 known solutions using the same strip. If you run out of pH paper cut another small strip.
9. Dispose of the pH paper in the regular trash can.

Part D: Precipitation and Reactivity Tests

1. Obtain a tray containing the following four test reagents: 0.2 M $AgNO_3$ (caution – can stain skin and clothes black, causes no harm), 0.2 M Na_2CO_3, 3 M NH_4OH (caution – caustic, keep away from skin and eyes), and Benedict's Reagent.
2. Record the observations for the glucose and isopropyl alcohol tests (step 3 below) in Experimental Data table 2.
3. Tests for glucose and isopropyl alcohol:

   Chromic acid tests for the presence of certain alcohols.[5] Benedict's reagent tests for the presence of certain sugars and alcohols.[6] Use these two tests on known solutions of glucose and isopropyl alcohol to see what a positive test looks like.

   a. Obtain two 13 mm x 100 mm test tubes. Label one as $C_6H_{12}O_6$ (B) or 1B. Label the other as C_3H_8O (B) or 2B.
   b. Transfer a portion of the glucose solution (test tube #1) into the test tube labeled $C_6H_{12}O_6$ (B) or 1B (see Figure 2) until it is approximately half-full.
   c. Transfer a portion of the isopropyl alcohol solution (from test tube #2) into the test tube labeled C_3H_8O (B) or 2B (see Figure 2) until it is approximately half-full.

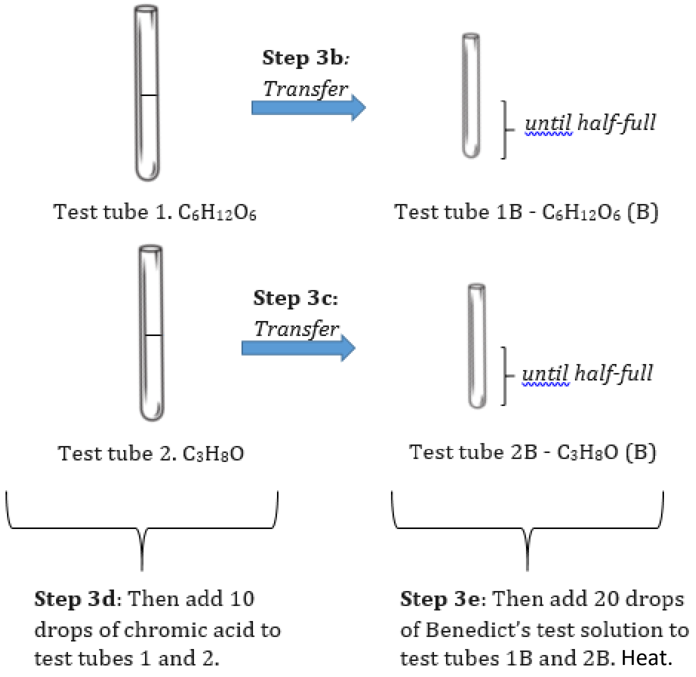


***Figure 2**. Tests for glucose and isopropyl alcohol.*

d. Chromic acid test:
   i. Add 10 drops of chromic acid to test tubes containing $C_6H_{12}O_6$ and C_3H_8O (#1 and #2 – the two taller test tubes; see Fig. 2). [Chromic acid is in the hood, take these two test tubes and place them in the test tube rack in the hood, carefully add the chromic acid (caution – caustic, keep away from skin and eyes, use gloves)].
   ii. A change in the color of chromic acid is evidence of a chemical reaction. Record observations.

GOLDEN LAB RULE:
Prevent contamination: hold coin stoppers between your fingers with the tip pointing downward while using the reagent.

e. Benedict's reagent test :
   i. Set up a hot water bath: <u>half-fill</u> a 250-mL beaker with DI water. Heat the water on a hot plate to a temperature between 60-70 ˚C.
   ii. Add 20 drops of Benedict's test solution to test tubes test tubes containing $C_6H_{12}O_6$ (B) and C_3H_8O (B) (1B and 2B – the two shorter test tubes; see Fig. 2).
   iii. Once the water bath reaches the targeted temperature range, place test tubes 1B and 2B in the water bath for about 5 minutes.

GOLDEN LAB RULE:
The temperature displayed on the hot plate is not the temperature (T)of the water. Measure T_{water} with thermometer.

        iv. Using a test tube holder, remove these test tubes from bath. Record observations.
    f. Discard the contents of the four test tubes containing glucose and isopropyl alcohol solutions in the Organic Waste Container located in the hood.

4. Tests for ionic compounds and acids:
   You will test for reactivity of the known solutions remaining (solutions #3 - #6) with the following three <u>test reagents</u>:
   - 0.2 M $AgNO_3$
   - 0.2 M Na_2CO_3
   - 3 M NH_4OH

   Record your observations for this section in Experimental Data table 3.
   a. Obtain 3 disposable pipets, and one 24-well plate. Wash the well plate and rinse it with three small portions of DI water; shake excess water over the sink (it is not necessary to dry it). Place a sheet of paper or marking tape above and next to the well plate and write the names or numbers of the solutions and the reagents. You can use a setup similar to the one shown in Figure 3.
      Note 4*: Several reactions produce white solids (observed as precipitates or cloudy solutions). It is easier to see them if the well plate is on the black tabletop instead of on a white sheet of paper.*
   b. Add 10 drops of acetic acid (test tube #3) to three wells in the first "row" using a disposable pipet.
      i. Add 10 drops of 0.2 M $AgNO_3$ to the first well containing acetic acid. (Be careful to only add drops to the desired well so that you do not cross-contaminate.) Record observations.
      ii. Add 10 drops of 0.2 M Na_2CO_3 to the second well containing acetic acid. Record observations.
      iii. Add 10 drops of 3 M NH_4OH to the third well containing acetic acid. Record observations.
      iv. Mix each well with glass rod, measure and record the solution's pH. Compare values to the pH of the acetic acid measured previously.
   c. Add 10 drops of hydrochloric acid (test tube #4) to three wells in the second "row" using a disposable pipet.
      - Repeat steps Part 4.b. i-iv above, this time with hydrochloric acid instead of acetic acid.
   d. Add 10 drops of magnesium sulfate (test tube #5) to three wells in the third "row" using a disposable pipet.
      - Repeat steps Part 4.b. i-iv above, this time with magnesium sulfate instead of acetic acid.
   e. Add 10 drops of aluminum chloride (test tube #6) to three wells in the fourth "row" using a disposable pipet.
      - Repeat steps Part 4.b. i-iv above, this time with aluminum chloride instead of acetic acid.
   f. Do not empty out the well plate yet. You will probably use it in Part E.

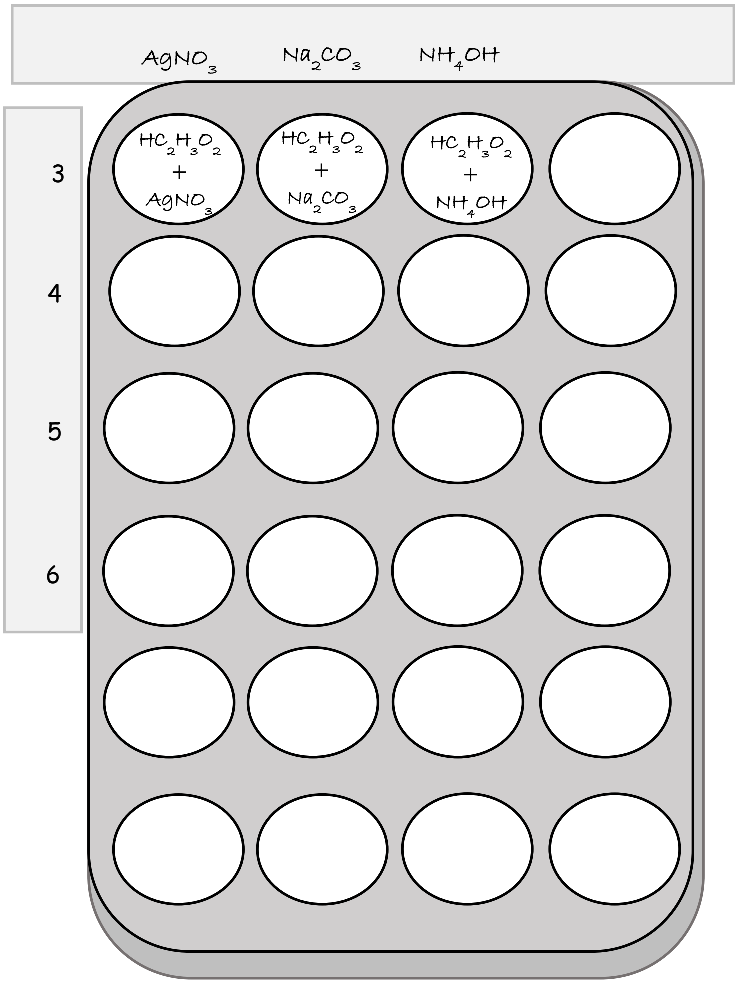


Figure 3. *Setup for reactivity tests using a 24-well plate. Chemicals listed on top border are the test reagents; the numbers on left side are the known solutions.*

Part E: Determination of the Identity of the Unknown Solution

1. Obtain a sample of the unknown solution located on the professor's lab bench or other designated area. Record its number in the designated area below Data Table 3.
2. Decide which tests done on the known solutions you want to repeat, this time on the unknown. Record the information in an organized manner. You can save time by thinking about the results of each test. For example, if you find that the unknown is not conductive, which reagent tests can you skip?
3. Based on the comparison of the results from the known solutions and the unknown solution determine and report the identity of the unknown solution.

Clean up/Disposal

- Discard the known solutions left in the test tubes containing acids and ionic compounds in the "Ionic/Acid Waste" container located in the hood.
- Discard mixtures in the 24-well plate in the "Ionic/Acid Waste" container in the hood.
- Discard the isopropyl alcohol and glucose in the "Organic Waste" container.
- Discard the unknown solution based on its identity, in the correct waste container as stated above.
- Discard the DI water down the sink drain.
- Discard disposable droppers in garbage can (first rinse them with water, transfer this waste water into the "Ionic/Acid Waste" container located in the hood).
- Wash and dry the outside of all glassware and return to original location.

Technique 1

Pre-lab

1. Classify the 6 known solutions as ionic compounds, molecular compounds, or acids.
2. A solution of 1.2 M chlorous acid, $HClO_2$, was found to have a pH of 3.
    a. Determine the concentration of the hydrogen ion. Report your answer to two significant figures (see page 5).
    b. Calculate the percent ionization of $HClO_2$.
    c. Classify $HClO_2$ as a strong or weak acid.
3. Using the solubility rules found in Appendix 3, organize the following compounds into two groups, water soluble and water insoluble: $Ba(NO_3)$, $CaSO_4$, $CoCO_3$, NH_4Br, $Sr(OH)_2$, $Na_2Cr_2O_7$, $Cu_3(PO_4)_2$, Fe_2O_3.
4. Solid sodium sulfate, Na_2SO_4, is water soluble. Write an equation showing how it dissociates in water.
5. A solution of iron(III) nitrate, $Fe(NO_3)_3$, was mixed with a solution of potassium carbonate, K_2CO_3. A precipitate was formed. What is the chemical formula of the precipitate? (See Appendix 2 and Appendix 3 at the end of this experiment.)

Post-lab

The laboratory report should include the following items:

1. All data tables.

2. For the two acids studied in this experiment:
   a. Calculate* the concentration of hydrogen ions in each solution.
   b. Calculate* the percent ionization.
   c. Classify each as a strong acid or weak acid.
   *Show calculations for at least one of them.
   d. Write an equation for the ionization of the two acids. Include phases.

3. Write the chemical formula for the 6 solids formed in Part D4 Tests for ionic compounds, and acids. Report this in the following manner:

Solids Formed from the Reaction of Known Solutions with Test Reagents

Chemical formula of Known Solution	Chemical formula of Test Reagent	Chemical formula of Solid Formed

4. Write a conclusion (see Appendix 9). Clearly state the unknown number and its identity.

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1. Tro, N. J. *Chemistry, A Molecular Approach*. 3rd ed.; Upper Saddle River, NJ, 2009; 159.
2. Holmquist, D. D.; Randall, J.; Volz, D. L. *Chemistry with Vernier*. Vernier Softwater & Technology: Beaverton, 2007; 13-1–13-3.
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VALENCIA COLLEGE

Experiment 3:
Electrolytes and Nonelectrolytes
Experimental Data and Calculations

Name: ____ **Date:** ____________________

Lab Partner: ____ **Section:** __________

Table 1. Conductivity and pH of the Known Solutions

Known Solutions	**Conductivity ( )**	**pH**
1) 0.50 M glucose ($C_6H_{12}O_6$)		
2) 50 % isopropyl alcohol (C_3H_8O)		
3) 0.50 M acetic acid ($HC_2H_3O_2$)		
4) 0.50 M hydrochloric acid (HCl)		
5) 0.50 M magnesium sulfate ($MgSO_4$)		
6) 0.50 M aluminum chloride ($AlCl_3$)		
7) Deionized water (H_2O)		

Table 2: Tests Results for Glucose and Isopropyl Alcohol Mixed With Several Test Reagents

Known Solutions	**Observations Upon Mixing**	
	Chromic acid	**Benedict's reagent**
1) 0.50 M glucose ($C_6H_{12}O_6$)		
2) 50 % isopropyl alcohol (C_3H_8O)		

Name: ______________________________

Table 3: Tests Results for Ionic Compounds and Acids Mixed With Several Test Reagents

Number and Identity Known Solutions	**Observations Upon Mixing**		
	0.2 M $AgNO_3$	**0.2 M Na_2CO_3**	**3 M NH_4OH**
3) 0.50 M acetic acid ($HC_2H_3O_2$)			
4) 0.50 M hydrochloric acid (HCl)			
5) 0.50 M magnesium sulfate ($MgSO_4$)			
6) 0.50 M aluminum chloride ($AlCl_3$)			

Analysis of Unknown Solution: Tests, Observations and Results

Unknown Solution Number ____________

Identity of Unknown Solution ______________

Name: ____

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VALENCIACOLLEGE

Experiment 4:
Limiting Reactant

Version 2a

Lynta Thomas, Ph.D. and Laura B. Sessions, Ph.D.

In this experiment, you will perform a chemical reaction varying the stoichiometry of the reactants to determine the limiting reactant and excess reactant in each trial. You will also calculate percent yield.

Objectives

- Assess chemical quantities related to compounds (mass, molar mass, moles, molecules/formula units, and percent composition).
- Understand mole relationships among reactants and products in a reaction given a balanced chemical equation.
- Evaluate and apply the concept of solution concentration.
- Calculate the theoretical yield and percent yield in a chemical reaction.

Learning Outcomes

- Understand the factors influencing chemical reactivity and quantitative relationships among species involved in a reaction.
- Employ conceptual learning outcomes and perform essential lab techniques in a laboratory setting.

Definitions

- **Actual yield** – the amount of product obtained in a chemical reaction
- **Coefficients –** numbers in front of the chemical formulas in a chemical equation that specify the relative amounts of each substance, in molecules or moles
- **Excess reactant** (or **excess reagent) –** the reactant that remains at the end of a chemical reaction when the limiting reactant is completed used up
- **Law of conservation of mass –** states that in a chemical reaction, matter is neither created nor destroyed
- **Limiting reactant** (or **limiting reagent) –** the reactant that is completely used up during a chemical reaction, and therefore limits the amount of product that can be formed
- **Molarity, M** – unit of concentration; expressed as moles of solute per liters of solution, mol/L
- **Percent yield –** a percent ratio of the product obtained (actual yield) divided by the theoretically possible amount of product (theoretical yield)
- **Stoichiometry –** the quantitative relationship between substances in a chemical reaction, based upon the law of definite proportions and the law of conservation of mass
- **Theoretical yield –** the amount of product predicted by the reaction stoichiometry

Techniques

- Technique 1: Cleaning Glassware
- Technique 2: Using a Balance
- Technique 5 Video Tech. 5: Using a Volumetric Pipet
- Technique 11: Disposing Chemical Waste
- Technique 17 Video Tech. 17: Filtration by Vacuum

Introduction

Metals are important materials due to their physical properties such as hardness, malleability, ductility, and high melting point.[1] They are used for many structural applications such as ships, buildings, underground tanks, and pipelines. One downside is that metals can be reactive and undergo corrosion especially in the presence of electrolytes (Figure 1); note the white color of the corrosion.[2] In one interesting example, an aluminum tank truck used to transport molasses suddenly showed corrosion, even though molasses does not normally cause this issue.[3] After extensive investigation, it was discovered that one batch of molasses had been made in a copper kettle. And, there were enough copper ions in the molasses from the kettle to cause corrosion of the aluminum tank.

Today, you will act as a chemical engineer to study the reactivity of aluminum metal in a solution of copper ions. Your supervisor at the Mole of Molasses Bulk Sweets Company would like a general report to share with your supplier of molasses, to try to convince them to discontinue the use of the copper kettles in order to save your tanker trucks.

Figure 1. A DC-6/C-118 Liftmaster Made of Aluminum and Fiberglass Shows Corrosion. "Aircraft Restoration" U.S. Air Force Photo by Tech. Sgt. Shawn J. Jones used under the Public Domain.

Chemical reactions are represented by balanced chemical equations. The equation provides a great deal of information about the reaction it represents and the substances involved. The amount of every substance consumed and produced in a chemical reaction is related to the amounts of all the other substances in the equation. According to the law of the conservation of mass, in a chemical reaction, there must be the same number of atoms of each kind on both sides of the equation. Since matter is conserved in a chemical reaction, chemical equations must balance for mass.

For example, consider the reaction between solid phosphorus and gaseous chlorine to produce liquid phosphorus trichloride. As represented by the chemical equation, the total mass of reactants equals the total mass of products.

$$\begin{array}{cccc} P_4(s) & +\ 6Cl_2(g) & \longrightarrow & 4PCl_3(l) \\ 123.90\ g & 425.40\ g & & 549.30\ g \\ 1\ mol & 6\ mol & & 4\ mol \end{array}$$

$$\begin{array}{llcl} reactants: & 123.90\ g & & products: \\ & \underline{+425.40\ g} & & \\ & \boxed{549.30\ g} & & \boxed{549.30\ g} \end{array}$$

The study of the numerical relationship between chemical quantities in a chemical reaction is called stoichiometry. The stoichiometric coefficients are the numbers in front of the chemical formulas in a balanced chemical equation. The coefficients specify the relative amounts of each substance, in molecules or moles, involved in the reaction. For example, when ethane is burned in the presence of oxygen, carbon dioxide, and water are produced:

$$2\ C_2H_6(l) + 7\ O_2(g) \longrightarrow 4\ CO_2(g) + 6\ H_2O(g)$$

According to the equation, 2 **molecules** of C_2H_6 react with 7 molecules of O_2 to form 4 molecules of CO_2 and 6 molecules H_2O, or 2 **moles** of C_2H_6 react with 7 moles of O_2 to form 4 moles of CO_2 and 6 moles H_2O.

The coefficients allow us to convert between numbers of moles of reactants and reactants, products and products, or reactants and products. The stoichiometric ratio is the ratio of the coefficients of any two species (reactants or products) in a balanced chemical reaction. Conversion factors can be set up to convert from moles of reactants to products or vice-versa using the stoichiometric ratio. For the reactant C_2H_6 and the product CO_2, the conversion factors are:

$$\frac{4\ moles\ CO_2}{2\ moles\ C_2H_6} \quad or \quad \frac{2\ moles\ C_2H_6}{4\ moles\ CO_2}$$

For chemical reactions with multiple reactants, it is likely that one of the reactants will be completely consumed before the others. A reactant that is completely consumed in a chemical reaction is called a limiting reactant or limiting reagent. When this reactant is completely consumed, the reaction stops and no more products are formed since the reaction cannot continue without it.

Returning to the example of ethane, we can ask how many grams of carbon dioxide can be made from 4.00 g of C_2H_6 and 6.00 g of O_2?

$$2C_2H_6(l) + 7O_2(g) \longrightarrow 4CO_2(g) + 6H_2O(g)$$

To solve, first convert mass to moles, since the stoichiometry is based upon moles or molecules:

$$4.00\ g\ C_2H_6 \times \frac{1\ mol\ C_2H_6}{30.070\ g\ C_2H_6} = 0.13\underline{3}0\ mol\ C_2H_6$$

$$6.00\ g\ O_2 = \frac{1\ mol\ O_2}{32.00\ g\ O_2} = 0.18\underline{7}5\ mol\ O_2$$

Note that extra digits are carried through the calculation to avoid rounding errors; the last significant figure is underlined.

Then, use the stoichiometry to determine which reactant will yield less product:

$$0.13\underline{3}0\ mol\ C_2H_6 \times \frac{4\ mol\ CO_2}{2\ mol\ C_2H_6} = 0.26\underline{6}0\ mol\ CO_2$$

$$0.18\underline{7}5\ mol\ O_2 \times \frac{4\ mol\ CO_2}{7\ mol\ O_2} = 0.10\underline{7}1\ mol\ CO_2$$

In this case, the oxygen is the reactant that makes the least amount of product; it is the limiting reactant. The maximum amount of the product that can be produced in a chemical reaction is determined by the limiting reactant.

The amount of product that can be produced from the limiting reactant in a chemical reaction is called the theoretical yield of the reaction. For the example of ethane, the theoretical yield is determined by the oxygen since it is the limiting reactant:

$$0.10\underline{7}1\ mol\ CO_2 \times \frac{44.01\ g\ CO_2}{1\ mol\ CO_2} = 4.7\underline{1}3\ g\ CO_2$$

Actual yield is the amount of product obtained experimentally, which may differ from the theoretical for many reasons. The actual yield of the product is expressed as a percentage of the theoretical yield and is called the actual percent yield or just percent yield. For example, if we burn 4.00 g of C_2H_6 and 6.00 g of O_2, and carefully collect the carbon dioxide produced, we may only obtain 4.620 g.

$$\%\ yield = \frac{(Actual\ yield)}{(Theoretical\ yield)}\ x\ 100$$

$$\%\ yield = \frac{4.620\ g}{4.7\underline{1}3\ g}\ x\ 100 = 98.\underline{0}3 = 98.0\%\ yield$$

This lower than expected yield occurs in combustion reactions since carbon monoxide (CO) and soot are produced as by-products of the reaction. Percent yield is a unitless quantity. When calculating percent yield, both the theoretical yield and the actual yield must be in the same units. These yield units need not be only in grams; the amount can also be expressed in moles or volume.

In this experiment, one of the reagents will be in solution. So, the moles of reagent will be calculated from volume and a known concentration. For example, if 20.00 mL of 0.25 M copper(II) chloride solution were pipetted into a flask, the number of moles of copper(II) chloride in the flask would be:

$$20.00\ mL\ CuCl_2 \times \frac{1\ L}{1000\ mL} \times \frac{0.25\ mol\ CuCl_2}{1\ L\ solution} = 5.0 \times 10^{-3} mol\ CuCl_2$$

In this experiment, you will add aluminum foil to a known concentration of an aqueous solution of copper(II) chloride. The products of the reaction are solid copper, aqueous aluminum chloride, and water. This is a single displacement reaction, which is also classified as an oxidation-reduction reaction. Aluminum is more active than copper and will displace copper from the copper(II) chloride solution. A red precipitate of metallic copper will form.

$$2\ Al(s) + 3\ CuCl_2(aq) \longrightarrow 3\ Cu(s) + 2\ AlCl_3(aq)$$

Aluminum is a highly reactive silvery-white metal. Usually, the surface of the metal is covered with a very thin layer of oxide (white Al_2O_3) that forms rapidly in air, providing excellent corrosion resistance and protecting the metal from further attack by air. Aluminum does not show its true reactivity until the oxide layer is disturbed.[4] Adding concentrated hydrochloric acid disturbs this oxide layer, and also serves to increase the reaction rate due to the activity of the hydrogen ion.

The stoichiometric ratios of the reactants and the products are known from the balanced chemical equation. The molarity and volume of the copper(II) chloride solution will be given to you; by weighing the aluminum, the limiting reactant in each experimental trial can be determined. Once the limiting reactant is identified, the theoretical yield of the reaction can be calculated. From the experiment results, the actual yield and percent yield of the precipitate formed is determined. You can expect high yield for this reaction (>90%). Make careful observations though since residual aluminum can cause elevated yields, and black copper(II) oxide can be a by-product.

Experimental Procedure

List of Chemicals

- ~0.50 M copper(II) chloride solution
- aluminum foil
- 6 M hydrochloric acid

Hydrochloric acid is corrosive to skin, eyes, and clothing. Copper(II) chloride is toxic via ingestion; be careful to wear gloves, avoid touching face, phone, and other items. Wear appropriate personal protective equipment. Wash hands well before leaving the lab. Use Safety Data Sheet (SDSs) to learn about proper handling of these chemicals. (The SDSs are available in the laboratory or online at www.hazard.com.)

Safety

List of Materials and Glassware

- Three 100mL beakers
- Four 250-mL beakers (or two 250-mL and two 150-mL)
- One 15-mL volumetric pipet
- Pipet bulb
- Büchner funnel and side-arm flask
- Clamp to hold the side-arm flask
- Filter paper
- Two glass stirring rods
- Oven set at 115 °C (or storage for samples to air dry until next week)

Part A: Identifying the Limiting and Excess Reactant in the Chemical Reaction

In this experiment, you will complete two sets of reactions. Label the two sets of reactions as Trial 1 and Trial 2. You will use 15.00 mL of copper(II) chloride solution, but different masses of aluminum foil for each trial.

1. You will be given a ~0.50 M copper(II) chloride stock solution. In your data table, record the exact concentration of the copper(II) chloride solution written on the label of the bottle. You will need this concentration to do your calculations.

2. Obtain three clean, dry 100-mL beakers.
3. Pour 50 mL of the copper(II) chloride solution into the first beaker. Label the beaker with the name of the solution.
4. Pipet 15.00 mL of copper(II) chloride solution into the second and third beakers. Label the beakers (Trial 1 and Trial 2). Remember to condition the pipet!

Technique 5
Video Tech. 5

5. Using an analytical balance, cut a piece of the aluminum foil with a mass between 0.09 and 0.11g. Write the actual mass of the aluminum foil displayed on the balance in your data table. This piece of aluminum foil will be used in the reaction labeled Trial 1.

Technique 2

6. Next, using the same analytical balance, cut another piece of the aluminum foil with a mass between 0.135 and 0.145g. Write the actual mass of the aluminum foil displayed on the balance in your data table. This piece of aluminum foil will be used in the reaction labeled Trial 2.
7. You must perform the following part of the experiment in a fume hood or under a snorkel fume extractor:
    a. Cut each aluminum foil in small pieces (to fit into the beaker and increase the reaction rate). Place each sample of the foil into the corresponding beaker containing the copper(II) chloride solution. Note the time.
    b. Record the initial color of the solution in each beaker. Note any observations in your data table.
    c. For the next ten minutes, stir the solution in each beaker every minute, using two glass stirring rods. Break up the chunks of copper forming with the stirring rod to allow all the aluminum to react.
    d. Measure 2.0 mL of 6 M HCl in a 10-mL graduated cylinder. Add the acid to the beaker labeled Trial 1 and stir the solution. Measure another 2.0 mL of the 6M HCl acid, and add to the beaker labeled Trial 2.
    e. Continue stirring both reaction mixtures regularly. Add 2.0 mL more of the HCl at 10 minute intervals until the reaction is complete.
    f. The reaction is complete when no more bubbles or effervescence are observed, and all the aluminum disappears.

While waiting, use this time to have one group member stir while the other sets up the filtrations for Part B.

8. Allow the reaction mixture to settle. Record the final color of the solution in each beaker. Predict the limiting reactant (Al or copper(II) chloride solution) in each trial, by observing the color of the solution in each beaker. Record all observations in your data table.
9. Compare your visual observations and the results obtained for the calculated values when determining the limiting reactants.

Part B: Filtration and Measuring the Product

You will perform vacuum filtration to separate the copper product.

Technique 17
Video Tech. 17

1. Obtain and label two 150-mL or 250-mL beakers (Trial 1 and Trial 2 and your initials). Mass each one. Record the masses in your data table.
2. Obtain two filter papers. Label outer edge of the filter papers using a pencil (Trial 1 and Trial 2 and your initials). Record the masses in your data table.
3. Now, use a vacuum filtration set-up to filter the Trial 1 solution in two steps: first the liquid, then the solid.
    a. Before transferring the solid onto the filter paper, pour only the supernatant solution from the beaker onto the filter paper using a glass stirring rod to direct the flow.
    b. Add 15 – 20 mL of distilled water into the beaker containing the solid copper and stir using the glass rod (to remove any excess acid trapped in the precipitate.) Allow the solid to settle and transfer the supernatant solution from the beaker onto the filter paper.
    c. Complete each filtration by transferring the entire solid onto the filter paper.
    d. Finally, thoroughly rinse the sides of the filter paper and the residue (red-colored solid copper) in the filter paper with distilled water.
    e. Leave the residue on the filter paper until all the water is completely drained.
4. Transfer each filter paper containing the copper from Büchner funnel to the corresponding beaker (labeled Trial 1 and Trial 2).
5. Heat beakers with the filter paper and solid copper for 15–20 minutes in an oven at 115 °C to dry them.
6. Using the tongs and hot gloves, remove the heated beakers containing the filter paper with the solid copper from the oven. Place each on a hot pad to cool.
7. After cooling, weigh each beaker with the filter paper and the solid copper. Record the masses in your data table.
8. Place each beaker with the filter paper and the solid copper back in the oven and continue heating for 5 more minutes. Allow it to cool, and re-mass. Continue heating, cooling, and weighing (two more times if required), until you get a constant mass (within ± 0.01 g) for the beaker with the filter paper and the solid.

The constant mass obtained after the heating and cooling is used for calculating the final mass of the solid product formed in the reaction. This mass is your experimental or actual yield. Note: It is incorrect to take the average of all the masses measured while drying. The average will not give you the accurate mass of the final solid product. Use the last mass only.

Clean up/Disposal

Technique 11

- Pour all liquid waste into the liquid waste container. Rinse the beaker and any glassware with small portions of water and discard the rinses in the liquid waste container.
- Solid waste must be discarded in the solid waste container.
- Clean the glassware with soap and tap water, and discard in the sink. Rinse it twice with distilled water, dry the outside of the glassware, and replace in its original location.
- Place the volumetric pipets tip up in the pipet canister.

Pre-lab

The pre-lab assignment must be completed before you come to the lab.

1. Given the chemical reaction: $Fe(s) + CuSO_4(aq) \rightarrow FeSO_4(aq) + Cu(s)$, answer the following questions.
   a. Is the chemical equation above balanced?
   b. Using the coefficients from the balanced chemical equation for the above reaction; write the mole-to-mole ratio between each of the reactants and solid product (copper.)
2. Write the balanced chemical equation for the reaction between solid aluminum and aqueous solution of copper(II) chloride.
   a. Using the coefficients from the balanced chemical equation for the above reaction; write the mole-to-mole ratio between each of the reactants and solid product (copper.)
   b. Determine the molar mass of aluminum metal and copper metal.
   c. Determine how many moles of copper(II) chloride are in 15.00 mL of a 0.500M solution.
   d. When 0.200 grams of Al reacts with 15.00 mL of a 0.500 M copper(II) chloride solution, how many moles of solid Cu would be produced? How many grams of solid Cu would be produced? Show all calculations including the mole-to-mole ratios used in the calculation.
3. If the solid copper product is recorded with the following information in the excerpt of Table 3 below, what should be recorded for the mass of copper product formed in the experiment (actual yield) in the last cell?

Table 3. Mass of Product

Trial 1	
Mass of empty beaker	29.570 g
Mass of filter paper	0.331 g
Mass of beaker + filter paper + copper product, after:	
First heating	30.254 g
Second heating	30.253 g
Third heating	30.253 g
Final mass of beaker + filter paper + copper product, after heating	30.253 g
Mass of copper product formed in the experiment (actual yield)	?

Post-lab

Show your calculations neatly for ***each*** trial as outlined by the tables 1-5. Also, write a conclusion (see Appendix 9 How to Write a Conclusion). Include:

1. A balanced chemical equation for the reaction in this experiment. Indicate the states of the reactants and products.
2. The limiting reactant and excess reactant in each trial. Back up your decisions with observations and calculations from the experiment.
3. Comment on your percent yield. Comment on any unusual or notable results. For example, if you received greater than 100 % yield, that would be an error since matter cannot be created; give some ideas for the source of the error. Likewise, give potential reasons for a very low yield, unexpected color changes, etc.

Experiment 4: Limiting Reactant
Experimental Data and Calculations

Name: ______________________________ **Date:** ________________

Lab Partner: ______________________________ **Section:** __________

Experimental Data and Calculations

Remember to include units of measure with each entry, and to read and record each measurement to the full precision allowed by the instrument used. Make your work neat and legible so that you may communicate your results to others. All tables should have a title; add a title where needed.

Table 1. Molar Mass of Reactants and Solid Product

1. Molar mass of copper(II) chloride	
2. Molar mass of Al metal	
3. Molar mass of Cu metal	

Table 2. Amount of Reactants Used in Each Trial

1. Trial #	Trial 1	Trial 2
2. Molarity of copper(II) chloride solution		
3. Volume of copper(II) chloride solution		
4. Moles of copper(II) chloride		
5. Mass of Al foil		
6. Moles of Al		

(Note: the clear cells should contain your data; the shaded cells will contain calculated values.)

Show your work for each type of calculation for each trial:

Table 3. Amount of Copper Produced in Each Trial

1. Trial #	Trial 1	Trial 2
2. Mass of empty beaker		
3. Mass of filter paper		
4. Mass of beaker + filter paper + solid product, after:		
a. First heating		
b. Second heating		
c. Third heating		
d. Final mass of beaker + filter paper + product, after heating		
5. Mass of solid copper formed in the experiment (actual yield)		

Show your work for each type of calculation for each trial:

Table 4. ______________________

1. Trial #	Trial 1	Trial 2
2. Moles of the product formed from copper(II) chloride		
3. Moles of the product that formed from Al		
4. Limiting reactant in the reaction (chemical formula)		
5. Excess reactant in the reaction (chemical formula)		
6. Theoretical yield in moles		
7. Theoretical yield in grams		
8. Theoretical mass of the unreacted excess reactant		

Show your work for each type of calculation for each trial:

Table 5. ____________________________

1. Trial #	Trial 1	Trial 2
2. Actual yield (mass of solid copper formed in the experiment)		
3. Theoretical yield		
4. Percent yield		

Show your work for each type of calculation for each trial:

Reporting to the Instructor

On completion of this experiment, show your calculations to your instructor.

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VALENCIACOLLEGE

Experiment 5:
Observing and Classifying Reactions

Version 2a

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Careful observations of eleven potential chemical reactions will allow determination of products and reaction classification for those that reacted.

Objectives

- Observe the characteristics of reactant substances, the characteristics of product substances, and energy effects to establish whether a chemical reaction has occurred.
- Classify reactions as combination (synthesis), decomposition, single replacement, or double replacement (metathesis).
- Write molecular equations for different types of reactions.
- Write complete ionic equations and net ionic equations.

Learning Outcomes

- Understand the nature of matter and its physical and chemical characteristics.
- Understand the nature and characteristics of chemical bonds.
- Understand and apply the rules of inorganic chemical nomenclature.
- Understand and apply the rules to write chemical formulas of ionic compounds.
- Perform essential lab techniques in laboratory setting.
- Write molecular, ionic, and net ionic equations.
- Balance chemical equations.

Definitions

- **Anion** – a particle with a net negative charge
- **Aqueous** – dissolved in water
- **Cation** – a particle with a net positive charge
- **Combination reaction** – see synthesis reaction
- **Complete ionic equation** – a chemical equation that lists all of the ions present individually
- **Compound** – a substance composed of two or more elements chemically bonded in a fixed, definite proportion
- **Decomposition reaction** – chemical reaction in which a single reactant breaks down into two or more products
- **Double replacement reaction** – chemical reaction where two compounds react, and the positive ions (cations) and the negative ions (anions) of the two reactants switch places, forming two new compounds or products
- **Electrolytes** – substances that conduct electricity when dissolved in water
- **Half-reactions** – chemical equations that show a portion of an oxidation-reduction reaction to include the electrons as either reactants or products

- **Ionic substance** – compound composed of cations and anions chemically bonded through electrostatic attraction
- **Ion** – atom or a group of bonded atoms with a net charge
- **Ionization** – process of gaining or losing electrons to become an ion
- **Law of conservation of mass** – a law that states that in a chemical reaction matter is neither created nor destroyed
- **Metathesis reaction** – see double replacement reaction
- **Molarity, M** – unit of concentration; expressed as moles of solute per liters of solution, mol/L
- **Molecular equation** – a balanced equation that shows complete neutral formulas for all compounds and their phases
- **Molecules** – a group of atoms bonded together, representing the smallest fundamental unit of a molecular compound (also applies to multi-atomic elements such as N_2 or S_8)
- **Moles** – a counting unit (a word that substitutes a counted amount), specifically the amount of material containing 6.022×10^{23} (Avogadro's number) particles
- **Oxidation** – process in which a reactant loses one or more electrons
- **Products** – chemicals formed or produced from a chemical reaction; found on the right side of the arrow in a chemical equation
- **Reactants** – starting chemicals involved in a chemical reaction; found on the left side of the arrow in a chemical equation
- **Reduction** – process in which a reactant gains one or more electrons
- **Single replacement reaction** – chemical reaction in which an element reacts with a compound and takes the place of another element in that compound
- **Solute** – the component in lesser amount in a solution
- **Solution** – a homogeneous mixture of a solute(s) and solvent
- **Solvent** – the major component of a solution
- **Stoichiometry** – the quantitative relationship between substances in a chemical equation, based upon the law of definite proportions and the law of conservation of mass
- **Spectator ions** – ions that do not participate in the reaction and therefore remain in solution as free ions
- **Strong electrolytes** – substances that dissociate/ionize in water completely or almost completely, forming ions; all ionic compounds and strong acids are strong electrolytes
- **Synthesis or combination reaction** – chemical reaction in which two or more reactants combine to form one single chemical compound
- **Weak electrolytes** – substances that dissolve in water but do not completely ionize and therefore only weakly conduct electricity in solution; weak acids and weak bases are weak electrolytes

Introduction

You have been awarded a conservation scientist internship at Laboratorio Scientifico dell'Opificio, in Florence, Italy. Conservation scientists are chemists that "help conservators understand materials and methods artists used to make works of art and, in some instances, they help determine what is original to the work and what was added in previous restorations. They also develop and test new materials - often from the chemical industry - to help conservators preserve artworks."[1]

Figure 1. Fresco "La Leggenda della Vera Croce", painted in the 15th century by Piero della Francesca before and after restoration using the Ferroni-Dini method. [2]

Molecular, Complete Ionic, and Net Ionic Equations

When an artist or restorer prepares art materials, such as grounds (a preparatory layer put on the support before a paint is applied), and paints, which consist of binders and pigments, they become immediately involved in chemical change. Chemical changes deal with changes in the structure of substances, the **reactants**. Bonds between atoms in reactants are broken and atoms are rearranged and bonded into new substances called **products**. In the process of chemical change, atoms are conserved. That is, all the atoms present in the reactants are present in the products, a requirement of the law of conservation of matter. A chemical change can be written as a chemical equation.

$$Na_3PO_4(aq) + 3AgNO_3(aq) \rightarrow Ag_3PO_4(s) + 3NaNO_3(aq) \qquad \text{Equation 1}$$

A **chemical equation** is simply a shorthand way of describing a chemical reaction. Equation 1 could be written out in a sentence that would read: "1 mole of aqueous sodium phosphate reacts with 3 moles of aqueous silver nitrate, to produce one mole of solid silver phosphate and 3 moles of aqueous sodium nitrate." The chemical equation says the same thing in a lot less space! If you examine this equation closely and count the number of atoms on each side of the arrow, you should see that this is also a **balanced chemical equation.**

Equation:	*Na*	*P*	*O*	*Ag*	*N*
Left side	3	1	13	3	3
Right side	3	1	13	3	3

A balanced chemical equation obeys the law of conservation of mass. It includes several important things:

1. The correct formulas for **reactants** and correct formulas for **products.**
2. An arrow to mean produces or yields.
3. The correct ratio (stoichiometry) in which the atoms, molecules, or moles of starting materials react with each other.
4. The correct stoichiometry of atoms, molecules, or moles of products formed.
5. Additional information might be included such as the substance's state written in parenthesis to the right of the substance (g - gas, l - liquid, s – solid, aq - aqueous) or special conditions placed above the arrow (Δ - heat, c - catalyst).

Equation 1 is also called a **molecular equation** because it shows complete neutral formulas for all the compounds.

Since aqueous ionic compounds and aqueous strong acids (Appendix 4) are strong electrolytes, they exist as virtually 100% dissociated in water. Therefore, a **complete ionic equation**, an equation that shows all the ions present, is a truer representation of the reaction. Equation 2 is the complete ionic equation for Equation 1.

$$3\ Na^{+}(aq) +\ PO_4^{3-}(aq) + 3\ Ag^{+}(aq) + 3\ NO_3^{-}(aq) \rightarrow\ Ag_3PO_4(s) + 3\ Na^{+}(aq) +\ 3\ NO_3^{-}(aq) \qquad \textit{Equation 2}$$

Notice that the three sodium ions and the three nitrate ions appear the same on both sides of the equation. Since they have not actually reacted, they are spectator ions.

A **net ionic equation** only includes the ions that actually react and the product(s) they form. Equation *3* is the net ionic equation for Equation 1.

$$PO_4^{3-}(aq) +\ 3Ag^{+}(aq)\ \rightarrow Ag_3PO_4(s) \qquad \textit{Equation 3}$$

To prepare for this internship, you need to recognize some general types of reactions, and how to write molecular, ionic, and net ionic equations. You also need to learn Italian, so get busy!

General Types of Reactions

We often classify reactions. Though there are many types of reactions, the most common types of reactions include: (1) synthesis or combination reactions, (2) decomposition reactions, (3) single replacement reactions, and (4) double replacement or metathesis reactions.

1. **Synthesis or Combination Reactions.** A synthesis reaction occurs when two or more chemicals react to form one single chemical compound.

    Metals often combine with non-metals to form compounds. Heating may be necessary to start reaction. Paleolithic artists used the product of Equation 4, MnO_2, as a black pigment[3]; it is also used as a glass additive to impart pink color to the glass.[4]

    $$2\ \mathrm{Mn(s)} +\ \mathrm{O_2(g)} \rightarrow 2\ \mathrm{MnO_2(s)} \qquad \textit{Equation 4}$$

    Nonmetals may combine with oxygen to form binary compounds called nonmetal oxides:

    $$S_2(s) + 3\ O_2(g) \rightarrow\ 2\ SO_3(g) \qquad \textit{Equation 5}$$

    Synthesis reactions are not limited to elements; compounds also participate in synthesis reactions. Nonmetal oxides (such as CO_2, SO_2, SO_3) are a serious hazard to artwork because they react with water to form acids. This combination reaction is illustrated in the formation of sulfuric acid in *Equation 6* below.

    $$SO_3(g)\ +\ H_2O(l) \rightarrow\ H_2SO_4(aq) \qquad \textit{Equation 6}$$

2. **Decomposition Reactions.** A decomposition reaction is a reaction in which a single reactant breaks down into two or more products.

   For example, a number of solid substances will decompose to form simpler substances when heated, as seen in Equation 7. Many Renaissance painters used $Cu(OH)_2$ as the pigment in pale blue paint. Many of these paintings now have black spots because the decomposition product of $Cu(OH)_2$, CuO, is black.

$$\mathrm{Cu(OH)_2} \rightarrow \mathrm{CuO(s)} + \mathrm{H_2O(g)} \qquad \textit{Equation 7}$$

3. **Single Replacement Reactions.** A single replacement reaction is a reaction in which an element reacts with a compound and takes the place of another element in that compound, as depicted in Figure 2.

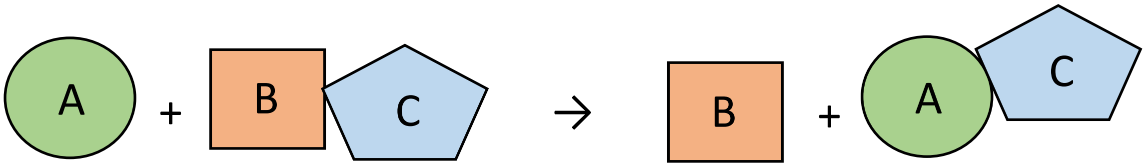


Figure 2. Representation of a Single Replacement Reaction

When a metal is placed in a solution containing ions of a second metal that is less reactive than it, the first metal donates electrons to the ions of the second metal in the solution and ions of the second metal will be converted to the free metal.

Equation 8a is an example of a single replacement reaction. The change in charge, which is easily observed in the net ionic equation (Equation 8b), is due to a transfer of electrons. Reactions in which electrons are transferred from one substance to another are called oxidation-reduction reactions or redox reactions.

$$Zn(s) + 2\,AgNO_3(aq) \rightarrow 2\,Ag(s) + Zn(NO_3)_2\,(aq) \qquad \textit{Equation 8a}$$

$$\mathrm{Zn(s)} + 2\,\mathrm{Ag^+(aq)} \rightarrow 2\,\mathrm{Ag(s)} + \mathrm{Zn^{2+}(aq)} \qquad \textit{Equation 8b}$$

To be able to "see" the electrons transferred, let's split Equation 8b into two half reactions, the oxidation half-reaction and the reduction half-reaction:

oxidation half-reaction $\quad Zn(s) \rightarrow Zn^{2+}(aq) + 2\,e^-$ *Equation 8c*

reduction half-reaction $\quad 2\,Ag^+ + 2\,e^- \rightarrow 2\,Ag(s)$ *Equation 8d*

Notice that Equation 8a and Equation 8b do not show the electrons because the number of electrons lost has to be the same as the number of electrons gained - in other words, electrons are transferred. It is necessary to have a coefficient of 2 in front of the silver metal and silver ion in Equation 8a and Equation 8b to allow the two electrons lost by zinc to be completely transferred since one silver cation only accepts one electron.

Metals more reactive than hydrogen will react with hydrogen ions in solution to produce hydrogen gas.

$$Zn(s) + 2\,HCl(aq) \rightarrow ZnCl_2(aq) + H_2(g) \qquad \textit{Equation 9}$$

4. **Double Replacement or Metathesis Reactions.** Many chemicals, when dissolved in water, form solutions containing positive and negative ions. If two such solutions are mixed, a reaction will occur if ions from the two solutions will combine with each other.

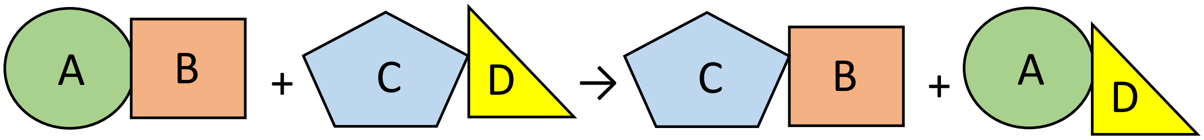


Figure 3. Representation of a Double Replacement Reaction

You will observe three types of double replacement reactions today:

Precipitation reactions: a solid or solids form. Equation 1 is an example of a precipitation reaction. The solubility rules for common ionic compounds in Appendix 3 can help you determine which ions will combine to form solids.

a. Formation of a gas, as seen in the example below.

$$Na_2S\,(aq) + 2HCl(aq) \rightarrow H_2S(g) + 2\,NaCl(aq) \qquad \textit{Equation 10}$$

Another example involves the reaction between sulfuric acid, H_2SO_4 (formed in Equation *6*) and the surface of frescoes. (A fresco is a painting done on the surface of wet, fresh plaster of calcium carbonate. Though origins date back to the 1st and 2nd centuries BCE in Greece, it was developed into a fine art during the Renaissance - 15th and 16th centuries CE. Perhaps the best-known example is the ceiling of the Sistine Chapel, painted by Michelangelo.) This reaction produces calcium sulfate, $CaSO_4$ and carbon dioxide. The calcium sulfate often crystallizes just beneath the surface and throughout the paint layer of frescoes causing severe blistering and loss of the fresco surface. The gas produced also affects the fresco surface. This reaction is shown in Equation 11.

$$CaCO_3(s) + H_2SO_4(aq) \rightarrow CaSO_4(s) + H_2O(l) + CO_2(g) \qquad \textit{Equation 11}$$

Examples of some common gases:

H_2S HCN NH_3 CO_2 SO_2 NO_2 NO

b. Neutralization reactions between an acid and a base produce water and an ionic compound. Acids cause changes in the color of many paints and degrade marble statues.

$$HC_2H_3O_2(aq) + NaOH(aq) \rightarrow NaC_2H_3O_2(aq) + H_2O(l) \qquad \textit{Equation 12}$$

Many weak electrolytes are acids and bases.

Examples of some weak electrolytes:

H_2O $HC_2H_3O_2$ H_3PO_4 HCN HF H_3BO_3 H_2SO_3 HNO_2 H_2S NH_4OH

When writing complete ionic and net ionic equations, keep weak electrolytes in a molecular form, as is observed in the exercise below, because weak electrolytes dissociate very little.

Exercise 1. Write the complete ionic and net ionic equations for the reaction between acetic acid and aqueous sodium hydroxide.

> Equation 12 is the molecular equation for the reaction between acetic acid and sodium hydroxide. Since acetic acid, is a weak electrolyte, it should not be dissociated in the complete ionic or net ionic equations.
>
> Ionic equation:
>
> $$HC_2H_3O_2(aq) + Na^+(aq) + OH^-(aq) \rightarrow Na^+(aq) + C_2H_3O_2{}^-(aq) + H_2O(l)$$
>
> Net ionic equation:
>
> $$HC_2H_3O_2(aq) + OH^-(aq) \rightarrow C_2H_3O_2{}^-(aq) + H_2O(l)$$

Techniques

The following techniques are used in the experimental procedure:

- Technique 1: Cleaning glassware
- Technique 8: Decanting
- Technique 9, Video Tech. 9: Using a Bunsen burner
- Technique 10: Using a centrifuge
- Technique 11: Disposing chemical waste
- Technique 12: Using pH paper
- Technique 15: Testing for Odors
- Splint test – explained in experiment


Technique

List of Chemicals

- *0.1 M ammonium chloride
- *0.2 M ammonium oxalate
- *0.1 M barium chloride
- *0.1 M calcium chloride
- solid copper
- *0.1 M copper (II) nitrate
- deionized (DI) water
- *1.0M hydrochloric acid
- **6.0 M hydrochloric acid
- *0.5 M silver nitrate
- *1.0 M sodium hydroxide
- **6 M sodium hydroxide
- *0.1 M sodium sulfate
- solid zinc

*Irritant to the skin, eyes, and mucous membranes. Avoid inhalation and other contact.


Safety

**Severe irritant to the skin, eyes, and mucous membranes. Avoid inhalation and other contact.

List of Equipment and Glassware

- one glass stirring rod
- one large beaker labeled as waste
- one test tube rack (if plastic, do not place a very hot test tube in it)
- twelve 13 mm x 100 mm test tubes (Once you use these, follow the steps listed in the Clean-up/Disposal section, and reuse them. They do not need to be dry inside.)
- one Bunsen burner
- one splint (a thin piece of wood)
- one test tube holder
- one medium size beaker (100-mL or 250-mL)
- centrifuge

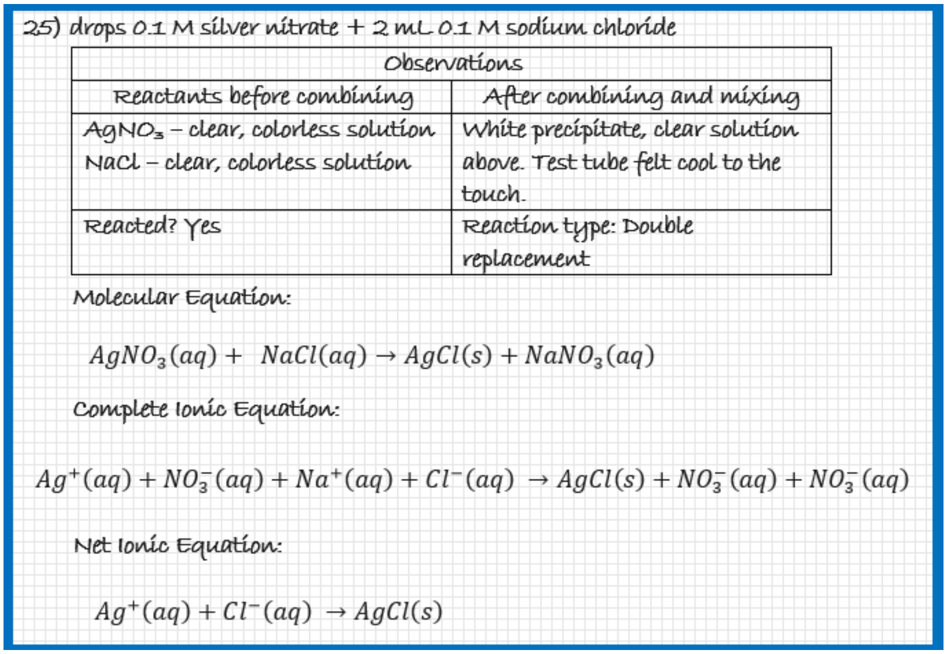
Experimental Procedure

Part A General Instructions

1. Be sure to carefully read the label on each chemical bottle. For example, Na_2SO_4 will not work if Na_2SO_3 is required. Check the concentration as well as the name. For example, the properties of concentrated (18 M) sulfuric acid are quite different from 3 M sulfuric acid.
2. Use 13 mm x 100 mm test tubes unless stated otherwise. Wash and rinse each test tube with deionized water. Dry the outside. These can be used wet, just shake to remove excess water.
3. When approximate volumes are needed, use the dropper on the bottles: 20 drops are approximately 1 mL.
4. Pour one reactant into the other - the order does not matter unless stated otherwise.
5. Mix all reactions thoroughly with a glass rod. After using the glass rod, rinse it with deionized water. Collect rinses in your waste beaker. No need to dry it before reusing.
6. Record all observations. Some observations to include (if applicable) are:
    - physical description: color, phase, hardness, crystallinity, shininess
    - temperature change (touch the test tubes before mixing and again after mixing; DO NOT TOUCH A TEST TUBE AFTER HEATING IT IN THE BUNSEN BURNER
    - gas (bubbling or effervescence is evidence of gas formation)
    - odor
    - solid (observed as a precipitate – bottom of test tube, or cloudiness – suspended in the solution)
    - any other observations you notice

Technique 15

7. Did a reaction occur? If so, write yes in the table, and classify the reaction based on the Reaction Types listed earlier in this experiment. If there is no reaction, then simply write no, and write a dash in the reaction type. Then move on.
8. If a chemical reaction occurs, write the molecular, ionic, and net ionic equations below as shown in Figure 4. Write these before moving on to the next reaction, unless stated otherwise. There is no need to try to write an equation if a reaction did not occur.



25) drops 0.1 M silver nitrate + 2 mL 0.1 M sodium chloride

Observations	
Reactants before combining	After combining and mixing
$AgNO_3$ – clear, colorless solution NaCl – clear, colorless solution	White precipitate, clear solution above. Test tube felt cool to the touch.
Reacted? Yes	Reaction type: Double replacement

Molecular Equation:

$$AgNO_3(aq) + NaCl(aq) \rightarrow AgCl(s) + NaNO_3(aq)$$

Complete Ionic Equation:

$$Ag^+(aq) + NO_3^-(aq) + Na^+(aq) + Cl^-(aq) \rightarrow AgCl(s) + NO_3^-(aq) + NO_3^-(aq)$$

Net Ionic Equation:

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$$

Figure 4. Recording Data and Equations – an Example

9. Some reactions ask for your instructor's signature. Have your instructor sign as soon as you complete the table and have written the equations, if applicable.
10. The following materials may be useful when writing the chemical equations:
    - Appendix 3: Solubility Rules
    - Appendix 4: Common Strong Acids and Strong Bases
    - Common gases listed after Equation 11
    - Some weak electrolytes listed after Equation 12

Part B. Reactions and No Reactions

Perform each reaction enumerated below. Record observations for each reactant in the corresponding Experimental Data Table. Then mix them together. Record observations and complete the table.

1. Reaction 1: Add 2 mL of deionized water into a 13 mm x 100 mm test tube. Then add a small amount (about half the size of a small pea) of zinc granules.
2. Reaction 2: Add 2 mL of 6.0 M hydrochloric acid into another 13 mm x 100 mm test tube. Place the test tube in a test tube rack. Since the reaction is fast, good coordination and team work is needed between you and your lab partner in the next steps involving the splint test:
    a. Turn on the Bunsen burner and ignite a splint (a thin piece of wood), blow out most of the flame (leave some embers that glow).
    b. Add a small amount (about half the size of a small pea) of zinc granules into the test tube containing the hydrochloric acid, and immediately place the tip of the splint with glowing embers, just inside the opening of the test tube. (If you do not detect a change, relight the splint, do not blow out the flame, and hold over the test tube. If you still do not detect a change, repeat the test with fresh reactants.)

Technique 9
Video Tech. 9

    c. Interpret the results of the splint test:
        - glowing splint will burst into flame when oxygen is present;
        - glowing splint will cause a small explosion (pop) when hydrogen is present; or
        - glowing splint will extinguish when carbon dioxide is present.
    d. Dispose of the splint in the trash can (wet it to ensure the flame is extinguished).
    e. Mix with glass rod. Observe closely: has the amount of solid zinc changed?

    Obtain instructor’s signature after you write the reactions, if applicable.

3. Reaction 3. Add a few drops of 0.2 M ammonium oxalate (the oxalate ion is $C_2O_4^{2-}$) to 1 mL of 0.1 M calcium chloride.

   Obtain instructor’s signature after you write the reactions, if applicable.

4. Reaction 4. Place a small piece of copper metal (turnings or wire) in 1 mL of 6.0 M hydrochloric acid. Observe the reaction when the wire is first placed in the solution, and then observe again several times for the next 10 to 15 minutes. (Continue with the next reactions; remember to come back and observe this reaction every so often).

   Obtain instructor’s signature after you write the reactions, if applicable.

5. Reaction 5. Place a small piece of copper wire in 2 mL of 0.5 M silver nitrate solution. Observe the reaction when the wire is first placed in the solution, and then observe again several times for the next 10 to 15 minutes. (Continue with the next reactions; do not forget to observe this one every so often). If the solution turns blue, this is evidence of the presence of Cu^{2+}.

   Obtain instructor’s signature after you write the reactions, if applicable.

6. Reaction 6. Add a few drops of 0.1 M barium chloride to 1 mL of 0.1 M sodium sulfate solution.
7. Reaction 7. Using graduated cylinders, place 2.0 mL of 1.0 M sodium hydroxide in one test tube and 2.0 mL of 1.0 M hydrochloric acid in another. Record how the temperature of the lower part of the test tubes feels to the touch, and measure and record the pH of each solution using pH paper. Pour the hydrochloric acid solution into the sodium hydroxide solution and swirl to mix. Record how the temperature of test tube feels to the touch and measure the pH of the mixture.

   Technique 4
   Technique 12

   A change in temperature or a change in pH of two or more pH units is evidence of a chemical reaction.

   (Note: it is sometimes difficult to deliver exactly the exact same amount; an extra drop of acid or base can affect the final pH observed.)

   Obtain instructor’s signature after you write the reactions, if applicable.

8. Reaction 8. Add 2 mL of 0.1 M ammonium chloride to 2 mL of 0.1 M copper (II) nitrate.

Part C. A Sequence of Reactions

Fill out the data table for each enumerated reaction below. Write equations if a reaction occurs.

9. Reaction 9. Add 6.0 M sodium hydroxide dropwise to 2 mL of 0.1 M copper(II) nitrate until no further reaction is observed. Save the products for the next step.
10. Reaction 10. Centrifuge the product from the step above. Decant the liquid. CAREFULLY heat the solid remaining in the test tube with a Bunsen burner. Keep the test tube moving in the flame, and keep the test tube out of the flame most of the time. Be sure that the tube is not pointing at anyone. Continue moving it in and out of the flame until the solid changes color. Save the product. In this reaction, one reactant makes two products: water in the gas phase and a

    Technique 10
    Technique 8

solid. (If the test tube rack is plastic or insulated metal wire, place the hot test tube in a medium size beaker while it cools.)

11. Reaction 11. Add 6.0 M hydrochloric acid dropwise to the solid produced in the step above. Stir after the addition of each drop. Add only enough hydrochloric acid to have a complete reaction.

Clean-up/Disposal

- Pour all solutions and solids into the inorganic waste container.
- Rinse each piece of glassware with deionized water, and collect the rinses into the inorganic waste container.
- Wash all glassware with soap and water, and then rinse with DI water. Dry the outsides of the glassware. Return all glassware to its place.

Technique 1
Technique 11

Pre-lab

1. The chemistry of fresco painting is the chemistry of limestone and lime plaster. Limestone is calcium carbonate ($CaCO_3$), an abundant, naturally occurring mineral. Two key reactions, shown below, are involved in the process of converting limestone to lime plaster (calcium hydroxide). Classify each of these reactions as one of the four **types of reactions** listed in this experiment (decomposition, synthesis, single replacement, or double replacement).
   a. $CaCO_3(s) \rightarrow CaO(s) + CO_2(g)$
   b. $CaO(s) + H_2O(l) \rightarrow Ca(OH)_2(aq)$
2. The Ferroni-Dini method was used to repair the damaged fresco shown in Figure 1. This method involves production of barium sulfate, a stable white solid, using the two-step process shown below. This solid was then dispersed using nanotechnology[5] to restore the fresco surface. Write **the complete ionic equation** for:
   a. $CaSO_4 \cdot 2H_2O(s) + (NH_4)_2CO_3(aq) \rightarrow (NH_4)_2SO_4(aq) + CaCO_3(s) + 2H_2O(l)$
   b. $Ba(OH)_2(aq) + (NH_4)_2SO_4(aq) \rightarrow BaSO_4(s) + 2NH_3(g) + 2H_2O(l)$
3. Balance the following equation:

   $$\_\,NaHCO_3(s) + \_\,H_2SO_4(aq) \rightarrow \_\,Na_2SO_4(aq) + \_\,H_2O(l) + \_\,CO_2(g)$$
4. A black precipitate was formed when 1 mL of 0.1 M $Pb(NO_3)_2$ was mixed with 1 mL of 0.1 M Na_2S.
   a. Write the chemical formula of the solid formed.
   b. Write the chemical formula of the spectator ion(s).
5. Two elements in their pure metallic form are used in this experiment. What are they?
6. Some solutions used in today's experiment are listed below. Write their chemical formula:
   a. ammonium oxalate
   b. calcium chloride
   c. copper(II) nitrate
   d. hydrochloric acid
   e. sodium hydroxide

Post-lab

- You do not need to write a conclusion for this experiment.
- The laboratory report should include the following items:
  a. Purpose
  b. Data tables for the 11 experiments.
  c. Molecular, complete ionic, and net ionic equations when applicable.

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VALENCIACOLLEGE

Experiment 5: Observing and Classifying Reactions

Experimental Data and Equations

Name: ________________________ **Date:** ____________

Lab Partner: ________________________ **Section:** ________

Purpose

Reactions and No Reactions

1. Deionized water + of zinc granules.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

Molecular Equation (ME):

Ionic Equation (IE):

Net Ionic Equation (NIE):

Name: ______________________________

2. 6 M hydrochloric acid + zinc granules. Perform splint test.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME:

IE:

NIE:

Instructor's Signature ____________________

3. 0.2 M ammonium oxalate (the oxalate ion is $C_2O_4^{2-}$) + 0.1 M calcium chloride.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME:

IE:

NIE:

Instructor's Signature ____________________

Name: ____

4. Copper metal + 6 M hydrochloric acid. Observe reaction several times for 10 - 15 minutes.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME:

IE:

NIE:

Instructor's Signature ______________________________

5. Copper metal + 0.5 M silver nitrate. Observe reaction several times for 10 - 15 minutes.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME:

IE:

NIE:

Instructor's Signature ______________________________

Name: ____

6. 0.1 M barium chloride + 0.1 M sodium sulfate.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME:

IE:

NIE:

7. 1.0 M sodium hydroxide + hydrochloric acid. Measure temperature and pH before and after.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME:

IE:

NIE:

Instructor's Signature ______________________________

Name: ______________________________

8. 0.1 M ammonium chloride + 0.1 M copper (II) nitrate.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME:

IE:

NIE:

A Sequence of Reactions

9. 6.0 M sodium hydroxide dropwise + 0.1 M copper (II) nitrate.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME:

IE:

NIE:

Name: ____

10. Centrifuge the product from experiment 9. Decant the liquid. Heat the solid.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME:

IE:

NIE:

11. 6 M hydrochloric acid + solid produced in experiment 10.

Observations	
Reactants before combining	After combining and mixing
Reacted?	Reaction type:

ME:

IE:

NIE:

VALENCIACOLLEGE

Experiment 6A
Amount of Active Ingredient in Aspirin

Version 1

Laura B. Sessions, Ph.D., Eileen Pérez, Ph. D., Diego J. Díaz López, Ph.D., and Anthony Mellone, Ph.D.

A sodium hydroxide solution will be prepared using serial dilution, and standardized by titration with hydrochloric acid. The sodium hydroxide will then be used to determine the amount of active ingredient in a commercial aspirin.

Objectives

- Perform a titration.
- Perform serial dilutions to obtain desired solution concentrations.
- Employ stoichiometric relationships and titration to determine an unknown quantity.

Learning Outcomes

Students will be able to:

- Apply proper rounding rules and rules of significant figures in calculations.
- Employ dimensional analysis (factor-label method) for unit conversions.
- Evaluate chemical quantities related to compounds (mass, molar mass, moles, and molecules/formula units).
- Identify and apply the concept of solution concentration.
- Apply the concept of serial dilutions.
- Prepare and complete labs to apply practical chemical concepts.
- Master essential laboratory techniques critical for laboratory science (titration and serial dilutions).
- Use scientific reasoning skills (such as observing, measuring, inferring, and predicting) to solve a solution to a simulated real-world situation.

Definitions

- **Acid-base reaction** – see neutralization reaction
- **Acids** – molecular compounds that form hydrogen ions, H^+, in water
- **Analyte** – the component of the sample to be studied or measured
- **Bases** – substances that form hydroxide ions, OH^-, in water
- **Concentration** – a measure of the amount of solute compared to solvent, often reported in molarity, M (mol/L), moles of solute in liters of solution

- **Endpoint** – when the indicator changes color during a titration; it is an estimate of the equivalence point, but often a little different (In this reaction, the indicator, phenolphthalein, turns pink in the presence of base so the equivalence point must be passed to have excess NaOH lead to color change.)
- **Equivalence point** – the stoichiometric point of the reaction, where the reagents have reacted completely
- **Indicator** – a substance that gives a visible change to show the presence or concentration of a chemical species
- **Neutralization or acid-base reaction** – when acid reacts with a base to form water and a salt
- **Standard solution** – a solution where the concentration is known to a great precision
- **Stock solution** – a solution that has a higher concentration than needed, used to prepare working solution(s) by serial dilution(s)
- **Titrant** – reagent used to carry out a titration, often the solution of known concentration
- **Titration** – process in which a solution of known concentration is reacted completely with a chemical of unknown amount
- **Working solution** – a solution prepared from the stock solution that we want to use in the experiment

Introduction

How do we know if medicines actually have the active ingredients that they are supposed to? Often times, we cannot detect if a medicine is present or working - certainly not by looking at it. The U.S. Food and Drug Administration (FDA) has Current Good Manufacturing Practices that create standards for ensuring pharmaceutical quality. By following the standards, the identity, strength, quality, and purity of drug products is assured. However, it is up to each manufacturer to choose how the standards are implemented. In addition, it is up to each manufacturer to report their compliance with the standards. The FDA does very little testing itself. It only reviews the results provided by pharmaceutical companies.

You have just been hired as a quality control expert for Pain Be Gone, a pharmaceutical company that makes aspirin. You need to check the latest lot of your company's aspirin to verify the amount of active ingredient. How would you measure active ingredients in a commercial aspirin?

Figure 1. Old Newspaper Advertisement of Bayer Aspirin.
Photo credit: Ads in History: 1926 Bayer Aspirin[1]*, used under the Creative Commons license.*

Aspirin

Aspirin, also known as acetylsalicylic acid, is a medication taken to temporarily relieve pain and fever. It has been promoted as pain reliever for a wide range of maladies as can be seen in the label depicted in Figure 1. Acetylsalicylic acid (see Figure 2) is a weak acid. Recall that **acids** are molecular compounds that produce hydrogen ions, H^+, in water (pH is a measurement of the concentration of hydrogen ion).

(a) $HC_9H_7O_4$

(b)

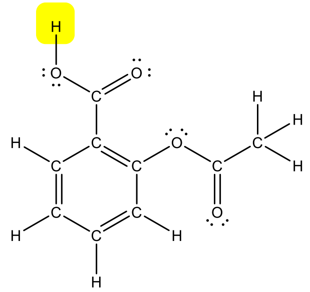

Figure 2. Acetylsalicylic acid.
(a) Chemical formula.   (b) Structural formula. (The acidic hydrogen has been highlighted in yellow.)

When ingested, acetylsalicylic acid remains intact in the acidic stomach but in the basic medium of the upper intestinal tract, it forms the salicylate and acetate ions.[2] Aspirin selectively inhibits the production of cyclo-oxygenase 1 and cyclo-oxygenase 2, which are enzymes responsible for inflammation and pain[3]. (It is interesting to note that historical accounts related to treating illnesses by ingesting extracts of salicylate-containing plants date back thousands of years. For example, about 2400 years ago, Hippocrates recommended treating eye diseases and pain with juices of poplar tree and willow bark, respectively, both of which are salicylate-containing plants.[4])

Different strengths of aspirin are based on the amount of acetylsalicylic acid present. In this experiment, you will measure this amount by using a quantitative technique known as titration. The process of titration allows determination of the concentration of an unknown acid or base by adding the exact amount of a base or acid to neutralize the other. A **neutralization** or **acid-base reaction** happens when an acid reacts with a base to form water and a salt. The balanced molecular equation for the reaction of acetylsalicylic acid and sodium hydroxide is shown in Equation 1 and in Figure 3.

$$HC_9H_7O_4(aq) + NaOH(aq) \rightarrow NaC_9H_7O_4(aq) + H_2O(l) \qquad \textit{Equation 1}$$

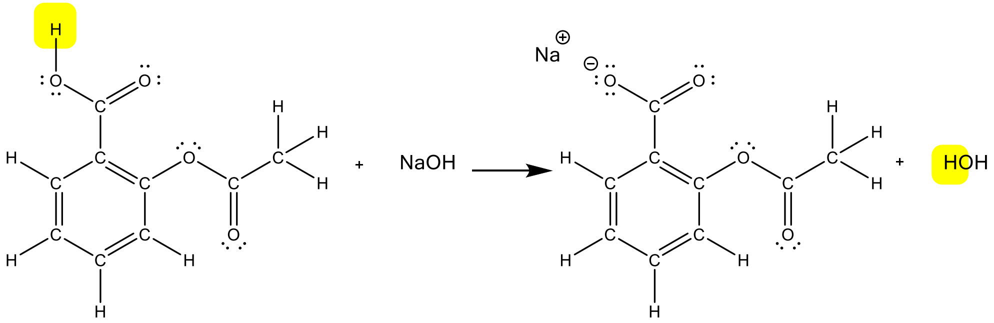

Figure 3. Structural representation of the reaction of acetylsalicylic acid with sodium hydroxide.

Titration

To determine the amount of acetylsalicylic acid present in an aspirin tablet, the easiest process would be to dissolve a tablet and titrate it with a base. **Titration** uses a solution of known concentration to react completely with an analyte to determine its concentration or amount. Titrations are usually set up with the solution of known concentration in a buret as seen in Figure 4 (though in Part A of this experiment the solution of unknown concentration will be in in the buret). The solution of unknown concentration, the analyte, is usually placed in an Erlenmeyer flask. Therefore, you might dissolve the aspirin tablet in water and slowly add 0.200 M NaOH to it until you reach the stoichiometric ratio, the **equivalence point** of the reaction. The **endpoint** of the titration, is shown with an indicator that changes color. From the known concentration and its volume used to reach the end point, the moles of the known solution are calculated, then the mole-to-mole ratio from the balanced reaction is used to determine the moles of the active ingredient. Finally, the mass of the active ingredient is found from its moles by use of its molar mass. Exercise 1 below, illustrates this process.

Buret: contains **titrant**, the solution of known concentration.

Graduations allow precise measurement of the volume added.

Erlenmeyer flask: contains **analyte**, the solution of unknown concentration.

Indicator is added to show the endpoint of the titration with a color change.

Figure 4. Components of a Titration

Exercise 1: A solution of magnesium hydroxide was titrated with a hydrochloric acid solution. What was the mass of $Mg(OH)_2$ in the solution if the titration required 71.10 mL of 0.150 M HCl?

The balanced equation for this reaction is:

$$Mg(OH)_2(s) + 2\ HCl(aq) \rightarrow MgCl_2(aq) + 2\ H_2O(l)$$

Solution:

a) Find moles of HCl that reacted by using the molarity equation:

Concentration is usually reported in molarity, M:

$$M = \frac{amount\ of\ solute\ (in\ mol)}{volume\ of\ solution\ (in\ L)} \qquad \text{Equation 2}$$

Rearranging equation 2 to solve for moles:

$$?\ moles\ HCl = M_{HCl} \times Volume\ HCl_L = \frac{0.150\ mol}{L} \times 0.07110\ L = 0.010\underline{6}65\ mol\ HCl$$

b) Find mass of $Mg(OH)_2$:

$$?\,g\ Mg(OH)_2 = 0.010\underline{6}65\ mol\ HCl \times \frac{1\ mol\ Mg(OH)_2}{2\ mol\ HCl} \times \frac{58.326\ g\ Mg(OH)_2}{1\ mol\ Mg(OH)_2} = 0.31\underline{1}023 = 0.311\ g$$

Titrating the Aspirin

There are a few steps that will lead to analysis of the aspirin. First, you need to prepare a known concentration of sodium hydroxide. To do this, you will be given a stock solution of approximately 1.6 M NaOH. You will dilute it to approximately 0.1 M NaOH and then standardize the diluted solution by titration with hydrochloric acid (HCl). A standard solution of HCl (approximately 0.15 M) will be provided; its exact concentration will be written on the carboy or bottle. (A standard solution is a solution whose concentration is known with a high precision.) Finally, you will dissolve the aspirin and titrate it with the NaOH solution you prepared to determine the amount of acetylsalicylic acid per tablet.

These steps lead to the following series of chemical and mathematical equations.

1) Dilute NaOH: for two solutions 1 and 2,

$$M_1 \times V_1 = M_2 \times V_2 \qquad \textit{Equation 3}$$

where: M is molarity
V is volume, in this equation it can be in L or mL as long as both volumes are in the same unit

2) Standardize NaOH using standard HCl:

$$\mathrm{NaOH(aq) + HCl(aq) \rightarrow H_2O(l) + NaCl(aq)} \qquad \textit{Equation 4}$$

3) Titrate the acetylsalicylic acid in the aspirin with sodium hydroxide. See Equation 1.
4) Calculate the mass of acetylsalicylic acid present in the aspirin. See Equation 9.

The analyses will be performed in triplicate. Report the mean and standard deviation using correct units and correct significant figures. Average or mean ($\bar{x}$) can be calculated by adding together values for all trials and dividing by the number of trials, as seen in Equation 5. Standard deviation will measure precision by showing the variation in a set of values around the mean. Equation 6 below has the formula for standard deviation.

$$\text{Mean:} \quad \bar{x} = \frac{x_1 + x_2 + x_3}{n} \qquad \textit{Equation 5}$$

$$\text{Standard deviation:} \quad s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}} \qquad \textit{Equation 6}$$

where: n is the number of trials
x_i is the individual result of each trial

Note: Standard deviation is usually reported to the same decimal places and unit of measure as the mean.

In addition, you will calculate the relative standard deviation (RSD, see Equation 7), and the relative percent error (see Equation 8).

$$RSD = \frac{s}{\bar{x}} \times 100 \qquad \text{Equation 7}$$

(If RSD<5%, you were precise in your measurements.)

$$Relative\ \%\ error = \left|\frac{true\ value - experimental\ value}{true\ value}\right| \times 100\% \qquad Equation\ 8$$

where: *true value* is the expected or theoretical value
experimental value is the calculated value based on data.

If relative % error ≤ 5%, the amount of active ingredient is acceptable.

Techniques

- Technique 1: Cleaning glassware
- Technique 3: Transferring liquids
- Technique 5, Video Tech. 5: Using a volumetric pipet
- Technique 6: Using a volumetric flask
- Technique 11: Disposing chemical waste
- Technique 16, Video Tech. 16: Filtration by gravity
- Technique 22, Video Tech. 22: Titration
- Technique 2: Using a balance
- Technique 4: Using a graduated cylinder
- Technique 23: Fan-Folding Flat Filter Paper

Techniques

List of Chemicals

- 1.6 M NaOH
- 0.10 M NaOH
- 0.15 M HCl
- Phenolphthalein indicator
- 95% Ethyl alcohol
- Commercial Aspirin, several brands

List of Equipment and Glassware

Week 1:

- two 50-mL beakers
- one 25-mL volumetric pipet
- one 15-mL volumetric pipet
- pipet bulb
- one 50-mL volumetric flask
- one 200-mL volumetric flask
- one 150-mL beaker
- one watch glass
- 3 Parafilm squares, or 3 watch glasses
- one magnetic stir bar
- one magnetic stirrer
- one buret
- one buret stand
- one buret clamp
- one small funnel
- one clean, dry plastic bottle
- three 125- or 250-mL Erlenmeyer flasks

Week 2:

- one mortar and pestle
- six 125-mL or 250-mL Erlenmeyer flasks
- three medium-sized funnels
- three fluted filter paper (or three flat filter papers)
- one 10-mL or 25-mL graduated cylinder
- 3 Parafilm squares, or 3 watch glasses
- one buret
- one buret stand
- one buret clamp
- one small funnel
- one magnetic stir bar
- one magnetic stirrer

Experimental Procedure

This experiment will take two weeks for completion. Part A will be done the first week. Report section for Part A is due at the beginning of week 2. Part B will be done the second week.

Part A Determination of NaOH(aq) concentration using standardized HCl(aq)

Procedure summary: You will prepare a working solution of NaOH (that you will label as NaOH Solution B) by performing serial dilutions. You will then determine the concentration of the NaOH Solution B by titration using as titrant the standardized HCl solution provided (see Equation 4).

Since the stock solution of NaOH is too concentrated, you will need to perform two serial dilutions. Use the NaOH provided to prepare a dilute NaOH Solution A, then use NaOH Solution A to prepare an even more dilute NaOH Solution B. NaOH Solution B is the working solution. Samples of NaOH Solution B will be titrated using the standardized HCl solution to determine its concentration. This procedure also prepares sufficient NaOH Solution B to perform the aspirin analysis lab.

1. **Prepare the NaOH Solution A and NaOH Solution B (serial dilutions).**
   Obtain about 45 mL of the stock solution of NaOH in a clean, dry 50mL beaker. Record its concentration, which is written on the carboy or bottle, on Data Table 1.
   a) NaOH Solution A: Using a 25-mL volumetric pipet (condition it first), deliver 25.00 mL of NaOH stock solution into a 50-mL volumetric flask. Fill the flask to the line with deionized water, cap, and invert to mix. Label the flask as NaOH Solution A.
   b) NaOH Solution B:
      i. Re-condition the above 25-mL pipet: Pour about 15 mL NaOH solution A into a dry 50-mL beaker. Rinse the pipet twice with deionized water, then rinse it twice with a few milliliters of the NaOH solution A that's in the beaker.
      ii. Place the pipet directly in the volumetric flask containing NaOH Solution A, fill it and deliver 25.00 mL of NaOH Solution A into a 200-mL volumetric flask.
      iii. Fill the 200-mL volumetric flask to the line with deionized water, cap, and invert to mix.
      iv. Label this flask as NaOH Solution B.

Technique 3
Technique 5
Video Tech. 5
Technique 6

2. **Prepare the HCl Standard samples that will be titrated.**
   a) Pour approximately 80 mL of the HCl Standard solution into a 150-mL beaker. Label and cover it with a watch glass. Record the concentration of the standardized HCl solution.
   b) Obtain three 125-mL or 250-mL Erlenmeyer flasks (if there are no more Erlenmeyer flasks available, use beakers), and a 15-mL volumetric pipet. Label the flasks as HCl Trial 1, HCl Trial 2, and HCl Trial 3.
   c) Using the 15-mL volumetric pipet (condition it first), deliver 15.00 mL of the HCl standard into each of these three Erlenmeyer flasks. Add 2-3 drops of phenolphthalein indicator. Cover with Parafilm or watch glasses, and set aside for now.

3. **Titration set-up.**
   a) Obtain a buret, a buret stand, buret clamp, a small funnel, a magnetic stirrer, and one magnetic stir bar.
   b) Place the buret clamp on the stand, and position the magnetic stirrer so that the buret may be suspended as close as possible over the center of the magnetic stirrer. See Figure 5.
   c) Condition the buret with the NaOH Solution B (use a funnel to pour the NaOH into the buret). Then add NaOH Solution B to the buret until the bottom of the meniscus is close to the 45-mL gradation mark. Suspend the buret with the clamp, and place the NaOH Solution B underneath it. Open the buret valve so the tip fills up with the solution. Do not allow it to completely empty out. Look closely just below the valve. If there is an air bubble, tap the buret while the valve is open, to see if it comes out. If not, ask your professor for help.
   d) Add NaOH Solution B into the buret until it is close to the 0 mL mark. Remove the funnel.

Technique 22
Video Tech. 22

Golden Lab Rule: Place funnel in a beaker or on a watch glass after use.

4. **Titrate the samples of HCl Standard using NaOH Solution B.**
   a) Add a magnetic stir bar into the HCl Standard - Trial 1 (Part A-2), place it on the magnetic stirrer, and turn the stirrer on so it mixes the solution quickly without splattering (or mix manually after each addition if not using the stirrer). Lower the buret until its tip is barely below the rim of the Erlenmeyer flask.
   b) Make the initial NaOH volume reading. Read the buret to a precision of ± 0.01 mL and record in Data Table 1.
   c) Start titrating: Add the NaOH into the HCl sample while continually mixing. As the endpoint nears, drip the NaOH solution into the HCl sample until the endpoint, which is when the solution turns pale pink and persists for at least 30 seconds. Stop titrating. Make the final NaOH volume reading. Read the buret to a precision of ± 0.01 mL and record in Data Table 1.
   d) Refill the buret with the NaOH Solution B. Repeat Step A4 with HCl Trials 2 and 3.

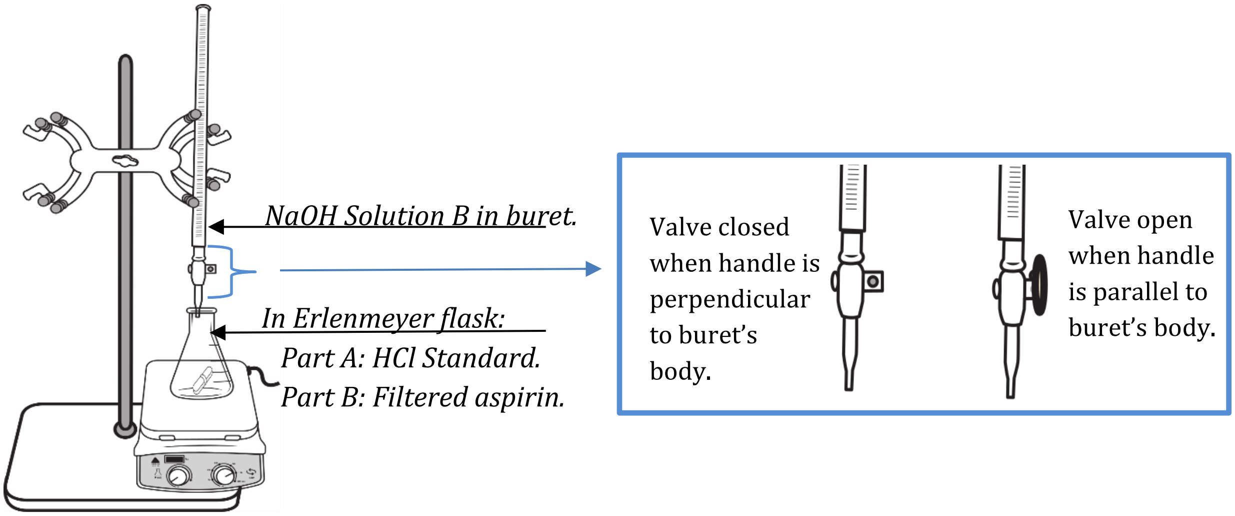

Figure 5. Titration setup (setup on left side of stand if left-handed).

5. **Save NaOH Solution B. The NaOH Solution B will be used for the aspirin analysis in the next lab period.**
   a) Transfer NaOH Solution B into a clean, dry plastic bottle. If the bottle is not dry, rinse it first with three small portions of NaOH Solution B. Close with cap.
   b) Using labeling tape, label the bottle as "NaOH Solution B", followed by the concentration (if you have not finished the calculations, write "Approximately 0.1 M"), write your names, and the date.
   c) Place in tray on professor's bench or other designated area.
6. **Calculations.**

   Calculate the concentration of NaOH Solution B determined by titration (the experimental value). Compare this experimental value with a theoretical value. The theoretical value of the NaOH concentration is calculated based on the NaOH stock solution's concentration reported on the carboy and the dilutions made to prepare Solution A and Solution B.

   a) Calculation of the concentration of NaOH Solution B (Theoretical value), and percent relative error.
      i. Calculate the concentration of NaOH in Solution A based on the concentration written on the carboy or bottle, the volume used, and the volume of NaOH Solution A prepared using Equation 3.
      ii. Calculate the concentration of NaOH in Solution B based on the concentration and volume of NaOH Solution A used and the volume of NaOH Solution B prepared using Equation 3.
   b. Calculation of the concentration of NaOH Solution B (Experimental value) in each trial.
      i. Calculate the moles of HCl Standard used in each Trial using Equation 2. Remember that the volume of the sample of HCl titrated was 15.00 mL.
      ii. Use the balanced equation, Equation 4, to calculate the moles of NaOH Solution B that reacted with the moles of HCl in each Trial.
      iii. Calculate the volume of NaOH delivered (final − initial buret readings).
      iv. Calculate the molar concentration of NaOH Solution B from each Trial (see Equation 2).
   c. Calculate the mean molar concentration of NaOH Solution B and its standard deviation using Equation 5 and Equation 6, respectively. Also, calculate the relative standard deviation using Equation 7.
   d. Finally, calculate the relative percent error between the mean experimental value and theoretical value for the concentration of NaOH Solution B using Equation 8.

Remember: these calculations are due at the beginning of the next laboratory period.

Part B Determination of the amount of acetylsalicylic acid in aspirin by titration with NaOH

1. **Prepare the aspirin sample Trial 1.**
   a. Select an aspirin brand. Record brand and amount of active ingredient on Data Table 2.
   b. Remove one tablet from the selected brand and weigh it. Record in Data Table 2 Trial 1.
   c. Pulverize the weighed tablet using a mortar and pestle.
   d. Tare a boat (or weighing paper) on the balance. Carefully transfer as much powdered sample into the boat as possible. Record the mass (record all digits from the analytical balance). You will need to determine the actual mass titrated in order to correct for the mass loss.

   Technique 2

   e. Transfer the weighed powder into a clean Erlenmeyer flask (125-mL or 250-mL). Rinse the boat with DI water over the flask to ensure the sample is completely transferred into the flask. Label the flask as Aspirin Trial 1.
   f. Using a graduated cylinder, add 10.0 mL of ethyl alcohol to the Erlenmeyer flask and stir for about 30 seconds with a glass rod. Then add 25.0 mL of water to the flask and stir again for another 30 seconds. The tablet will not completely dissolve due to inert ingredients (fillers and binding agents) in the tablet; the solution will be turbid. It will be necessary to filter it. (Remember to properly label your sample.)

   Technique 5
   Video Tech. 5

   g. Place a funnel in another clean Erlenmeyer flask (125-mL or 250-mL). See Figure 6a. (Note: If the funnel touches the bottom of the flask, suspend it above the flask using a support ring clamp and, if needed, a clay triangle (Figure 6b) held on a stand.)

   Technique 16
   Video Tech. 16

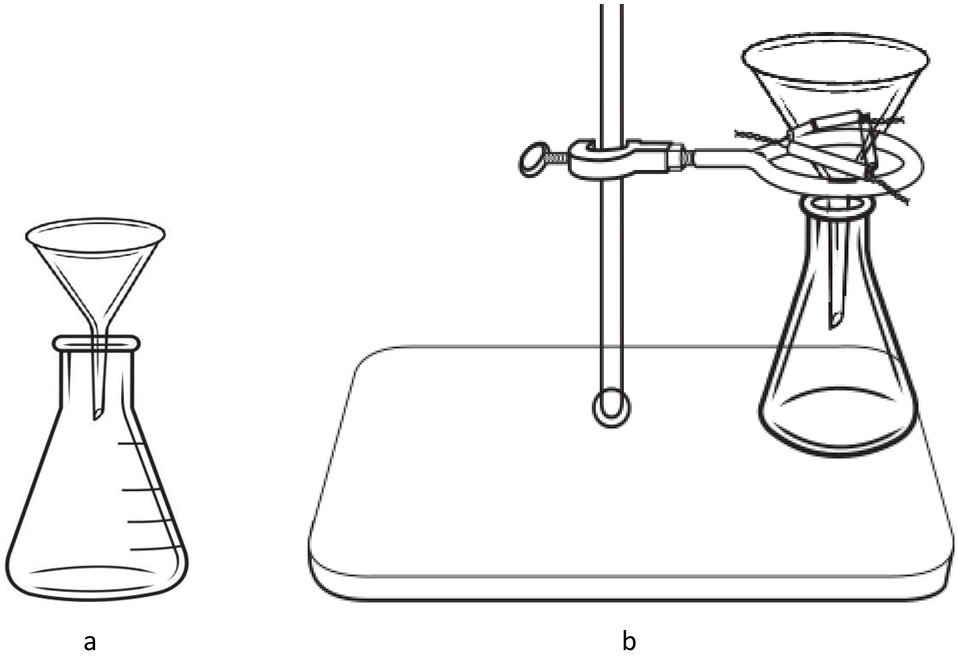

Figure 6. Funnel over Erlenmeyer flask

   h. Obtain a fluted filter paper (Fisherbrand Qualitative P8-Flute 09-790-14E) or fan fold a flat filter paper (Whatman #1 Qualitative). Open it (Figure 7).

Technique 23 video starting at 1:04

Figure 7. Open fluted (or fan-folded) filter paper

   i. Place the filter paper inside the funnel and seal it by wetting it with a small amount DI water. (You can leave this water in the Erlenmeyer flask.) Filter the aspirin sample prepared above. Once all the solution has gone through, rinse the filter paper and precipitate with a few milliliters of deionized water. Allow all the water to drain into the receiving flask. Rinse two more times.
   j. Remove funnel and cover flask with Parafilm or a watch glass.

2. **Prepare the aspirin sample Trials 2 and 3.**
   a. Prepare two more trials: obtain two additional tablets of the same aspirin brand, and repeat steps B1b – B1j; use a new filter paper and new flask each time. Label as Aspirin Trial 2 and Aspirin Trial 3.
3. **Titration set-up.**
   a. Retrieve the NaOH Solution B that you prepared last week (located on professor's bench or other designated area).
   b. Set up the titration following Part A steps 3a-3d.
4. **Titrate the aspirin samples.**
   a. Add 2-3 drops of phenolphthalein indicator and a magnetic stir bar to the filtered aspirin sample Trial 1. Lower the buret until its tip is barely below the rim of the Erlenmeyer flask.
   b. Make the initial NaOH volume reading. Read the buret to a precision of ± 0.01 mL and record in Data Table 2.
   c. Start titrating: Add the NaOH into the aspirin sample while continually mixing. As the endpoint nears, drip the NaOH solution into the aspirin sample until the endpoint, which is when the solution turns pale pink and persists for at least 30 seconds (titrate slowly – it takes longer for the aspirin sample to react with the NaOH near the endpoint). Stop titrating. Make the final NaOH volume reading. Read the buret to a precision of ± 0.01 mL and record in Data Table 2.
   d. Repeat Part B steps 4a-3c with aspirin Trial 2.
   e. Repeat Part B steps 4a-3c with aspirin Trial 3.

Technique 3
Technique 22
Video Tech. 22

5. **Calculation of active ingredient in the aspirin.**
   a. Calculate the volume of NaOH added and the moles of NaOH.
   b. Calculate the moles of acetylsalicylic acid that reacted with the NaOH in the titrated sample using Equation 1.

c. Calculate the grams of acetylsalicylic acid in the titrated sample using the molar mass acetylsalicylic acid.

d. Determine milligrams of acetylsalicylic acid (asa) in the <u>whole tablet</u> by using Equation 9.

$$mg_{\text{asa } per\ tablet} = mg_{\text{asa } in\ sample} \times \left(\frac{mass\ tablet}{mass\ pulverized\ sample}\right)$$

Equation 9

e. Calculate the mean $mg_{\text{asa } per\ tablet}$ and its standard deviation. Also, calculate the relative standard deviation.

f. Finally, calculate the relative percent error between the experimental mean and theoretical value (the value on label) for the $mg_{\text{acetylsalicylic acid } per\ tablet}$.

Clean up/Disposal

- Use a magnetic retriever (long plastic covered bar with a magnetic end) to remove magnetic stir bars. The retriever is usually hanging inside of the hood.
- Dispose all solutions into the waste container for liquids/aqueous solutions.
- Place pulverized solid tablet, and the filter paper with the precipitate in the waste container for solids.
- Rinse pipets and buret several times with small amounts of deionized water. Collect rinses into a waste beaker, transfer to waste container for liquids/aqueous solutions.
- Place pipets and buret, tip up, in the designated canisters.

Technique 11

Pre-lab

1. What volume, in milliliters, of a stock solution of 2.55 M NaOH would you have to use to prepare 1.00 L of a 0.500 M NaOH?

2. The titration of a 25.00 mL sample of NaOH required 28.25 mL of a 0.200 M HCl solution to reach the end point. What was the molar concentration of the sodium hydroxide solution?

3. A group obtained the following results:

Trial #	1	2	3
Amount active ingredient/tablet (mg)	598	583	587

Report the average, standard deviation and relative standard deviation (RSD) using correct units and correct significant figures.

4. A tablet of Pain Be Gone Aspirin, which had a mass of 1.213 g, was pulverized and 1.159 g were dissolved in 10.0 mL of ethyl alcohol and 25.0 mL of DI water. The titration of this solution with 0.1052 M NaOH required 15.62 mL to reach the phenolphthalein endpoint.

   Answer questions a – d below.

   a. Determine the moles of NaOH that reacted with the acetylsalicylic acid.
   b. Determine the mass, in grams, of acetylsalicylic acid in the sample analyzed.
   c. Determine the mass, in milligrams, of $HC_9H_7O_4$ in the tablet.
   d. The manufacturer claims that each tablet contains 325 mg ± 10 mg of acetylsalicylic acid. Is the actual amount of acetylsalicylic acid in the tablet acceptable?

Post-lab

In the data/calculations section:

1. Include the completed data tables.
2. In the calculations section show calculations requested.

Write a conclusion (see Appendix 9). Make sure to include the following in the conclusion:

1. Concentration of NaOH Solution B with its standard deviation.
2. The aspirin brand. The amount acetylsalicylic acid it should contain.
3. Properly report the experimental mean amount per tablet and its standard deviation.
4. The relative % error of the average mean amount per tablet.
5. State if the mean amount per tablet was acceptable or not. (Acceptable industry tolerance for this type of product ranges within 5% of the value stated on the label.)

References:

1. Hanneman, J. *Adds in History: 1926 Bayer Aspirin*. Treasured Lives [Online] May 1, 2016. https://treasuredlives.us/2016/05/01/ads-in-history-1926-bayer-aspirin/ (accessed July 3, 2018).
2. Beran, J. A. *Laboratory Manual for Principles of General Chemistry*, *10th ed.,* John Wiley & Sons: Hoboken, NJ, 2014; p 237.
3. Rainsford, K. D. Pharmacology and Biochemistry of Salicylates and Related Drugs. In *Aspirin And Related Drugs*, Rainsford, K. D. Ed.; CRC Press, Sheffield, UK, 2004; p 266.
4. Rainsford, K. D. History and Development of the Saicylates. In *Aspirin And Related Drugs*, Rainsford, K. D. Ed.; CRC Press, Sheffield, UK, 2004; p 2.

Experiment 6A: Amount of Active Ingredient in Aspirin
Experimental Data and Calculations

Name: ______________________ **Date:** ______________

Lab Partner: ______________________ **Section:** ________

Table 1. Determination of NaOH Concentration. (Part A due next Lab.)

1) HCl concentration written on carboy			
2) NaOH concentration written on carboy			
3) NaOH Solution A concentration (Theoretical value)			
4) NaOH Solution B concentration (Theoretical value)			
	Trial 1*	Trial 2	Trial 3
5) Volume of HCl sample			
6) Moles HCl			
7) Moles of NaOH			
8) Initial buret reading			
9) Final buret reading			
10) Volume NaOH added			
11) Molar concentration of NaOH Solution B (Experimental Value)			
12) Mean molar concentration of NaOH Solution B			
13) Standard deviation			
14) RSD			
15) Relative percent error (%)			

* Show calculations for Trial 1, standard deviation, RSD and relative % error. (Continue on back of sheet if needed.)

Name: ____

Table 2. Determination of Acetylsalicylic Acid in Aspirin.

1) Aspirin Brand (and amount of acetylsalicylic acid on label)			
2) Average molar concentration of NaOH Solution B (from Part A)			
	Trial 1*	Trial 2	Trial 3
3) Mass of aspirin tablet			
4) Mass of pulverized aspirin sample			
5) Initial buret reading			
6) Final buret reading			
7) Volume of NaOH Solution B added			
8) Moles of NaOH			
9) Moles of acetylsalicylic acid in sample			
10) Mass of acetylsalicylic acid in sample			
11) Amount of acetylsalicylic acid per tablet			

12) Mean amount of acetylsalicylic acid per tablet	
13) Standard deviation for acetylsalicylic acid per tablet	
14) RSD for acetylsalicylic acid per tablet	
15) Relative percent error (%)	

*Show all calculations related to Trial 1, standard deviation, RSD, and relative % error.

Name: ____

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Experiment 7
Analysis of a Gaseous Product

Version 2a

Eileen Pérez, Ph.D. and Tony Mellone, Ph.D.

A mixture containing calcium carbonate is reacted with hydrochloric acid. The carbon dioxide gas produced is captured and measured. A gas law and stoichiometric relationships allow for the determination of the percent of calcium carbonate in the mixture analyzed.

Objectives

- Determine the percent by mass of calcium carbonate in the garden lime under investigation.
- Measure pressure and become familiar with units of pressure.

Learning Outcomes

- Understand the nature of units of measurement and apply proper rules of significant figures to measurements and calculations.
- Understand and apply the relationships of physical behavior of gases.
- Enhance student's understanding of the application of the scientific method to solve complex problems.
- Understand and apply stoichiometric relationships.
- Understand and apply the relationships of physical behavior of gases.
- Perform essential lab techniques in laboratory setting.

Definitions

- **Alkaline** – a term referring to pH values above 7 when the solution is basic (related to the amount of hydroxide, OH^-, in the solution)
- **Analytical chemistry** – a field of chemistry that involves methods or instruments for separating, identifying, and determining amounts of compounds in a sample
- **Dalton's Law of Partial Pressure** – the total pressure of a gas mixture (P_t) equals the sum of the individual gas pressures ($P_t = P_A + P_B + P_C + ...$)
- **Environmental chemistry** – area of chemistry that focuses on chemical processes that occur in the environment and their impact on the environment
- **pH** – a scale that specifies the acidity of a solution; values typically run from 0-14, above 7 being basic, and below 7 being acidic
- **Relative percent error** – the absolute value of a calculation that indicates the accuracy of an experimentally determined value against the true, correct, or accepted value
- **Standard deviation** – a calculation that indicates the precision of a series of values by showing the variation around the average or mean
- **Volatile** – easily evaporates at normal temperatures

Introduction

The lawns of City Hall were destroyed. All the grass died in a matter of days. Upon investigation, the groundskeeper found that the lawn had been recently treated with Green-Thumb, Inc.'s garden lime. The pH of the soil was tested and found to be too high. For the last three years, City Hall has been using Green-Thumb, Inc.'s fertilizers, weed-killers, plant nutrients, pH soil kits, and garden lime. They have never had problems before this one! The Beautification Manager requested Green-Thumb, Inc. pay for the damages, but they refused. The company stands by their product and says that the formulation was correct. The Beautification Manager contracted lawyer Bergeron to sue the company for damages. The lawyer has subcontracted Dr. Nakamura, an environmental researcher from Valencia College, to analyze the garden lime in question. The groundskeeper has provided the lawyer with several bags of the garden lime. The label on the garden lime states that it is 70% calcium carbonate. Acceptable industry tolerance for this type of product ranges within ± 5% of the value stated on the label.

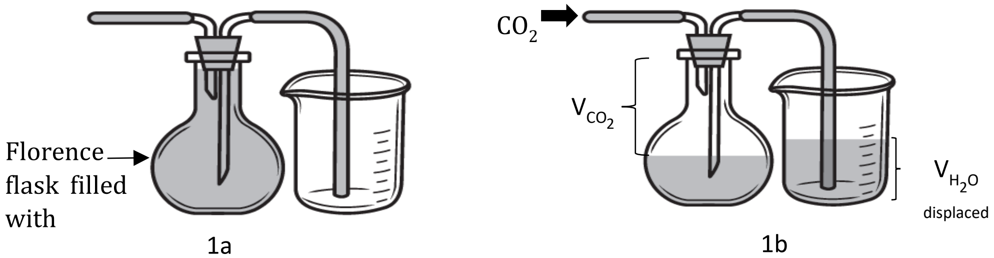
You are a graduate student pursuing your Ph.D. in analytical chemistry, doing research in Dr. Nakamura's lab. Since you have a master's degree in environmental chemistry, she has assigned your research team to determine the percent of calcium carbonate in the garden lime in question. As an environmental chemist, you know that the scale that measures pH runs from 0 to 14; neutral soil has a pH of 7. However, many factors can change pH from neutral including irrigation water, vegetation, and the parent material from which the soil is made. Above 7, soil is increasingly alkaline; below 7, it is increasingly acidic. Most plants grow best in slightly acidic soil with a pH of 6.5.[1] Garden lime is added to soils to reduce acidity; solid ground sulfur, iron(II) sulfate, or aluminum sulfate are added to reduce alkalinity.[1]

Garden lime is made from pulverized limestone[1]. Calcium carbonate (also called calcite) is the principal mineral found in limestone[1]. In addition to limestone, it is the principal mineral found in marble, chalk, pearls, stalactites, stalagmites, caliche, and the shells of marine animals such as clams.[2] It is also the principle mineral responsible for hard water. Calcium carbonate is an alkaline compound so it readily reacts with acidic media. Carbon dioxide is produced by this reaction:

$$CaCO_3(s) + 2\,HCl(aq) \rightarrow CaCl_2(aq) + H_2O(l) + CO_2(g) \qquad \textit{Equation 1}$$

The goal of this experiment is to determine the percent by mass of calcium carbonate in the ground garden lime under investigation. To accomplish this, you will treat a small sample of the ground garden lime mixture with an excess of 3 M hydrochloric acid, and collect the carbon dioxide gas produced. With proper measurements and stoichiometric relations, you will determine the percent of $CaCO_3$ in the sample.

- Determining the **volume** of CO_2 collected: If the CO_2 is collected in a flask containing water, the CO_2 will displace water equal to its volume (See Figure 1).



1a 1b

Figure 1. Florence flask used for gas collection.

- Determining the **pressure** of the CO_2 collected:
  Since water is volatile, the gas collected over water will be a mixture of two gases: water vapor and CO_2. Using Dalton's Law of Partial Pressure, the total gas pressure collected in this experiment will be:

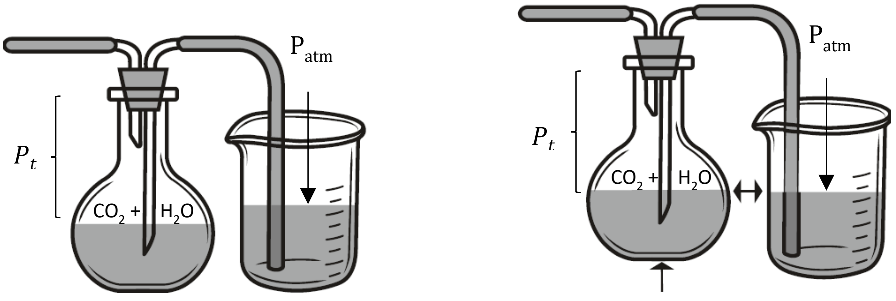
$$P_t = P_{CO_2} + P_{H_2O} \qquad \textit{Equation 2}$$

The atmospheric pressure of the room (P_{atm}) is equal to the total pressure in the flask, P_t, if the water level below the gas is physically held at the same height as that of the water displaced (see Figure 3). To understand the reason behind this, consider the U-tube illustrated in Figure 2. A U - tube is a hollow glass tube in the shape of the letter U. If the tube is empty and we pour mercury into the right arm (Figure 2a), when we stop pouring the mercury, the level of the mercury in the left arm will be equal to the level of the mercury in the right arm (Figure 2b). Why? Because both arms are open to the environment, therefore the pressure on both arms is equal.

2a 2b

Figure 2. Mercury poured into a u-tube.

Now consider Figure 3a. Since the height of the volume in the beaker is higher than the height of the volume in the Florence flask, we cannot assume that the external pressure (atmospheric pressure) is the same as the gas pressure in the flask. When the flask is lifted, as shown in Figure *3*b, the pressures are equalized. This causes a small change in the volume of the water displaced (that is why it is important to keep the hose below the water level in the beaker); volume and pressure are inversely proportional. This shift in liquid, though not detectable visibly since both the flask and beaker are wide, is desirable, because you want to know the amount of water displaced when P_t is equal to the atmospheric pressure. (Think of an old movie when a criminal places a hose into a car's gas tank valve and suctions: when the hose is lowered the gasoline gushes out, but when the hose is lifted the gasoline stops flowing.)



3a. $P_t \neq P_{atm}$ 3b. $P_t = P_{atm}$

Figure 3. Adjusting the pressure in the flask by raising the Florence flask.
(If the height of the volume in the beaker is lower, raise the beaker.)

The atmospheric pressure is measured using a barometer (it is probably located on the professor's demonstration bench). The vapor pressure of water is dependent on the temperature (see Appendix 5 Vapor Pressure of Water at Various Temperatures).

Following are some useful conversion factors involving pressure:

1 atm = 760 mm Hg = 760 torr = 29.92 inch Hg = 101.3 kPa

- Determining the **temperature** of CO_2 collected:
  Since the CO_2 is in contact with the water in the Florence flask, its temperature is the same as the temperature of the water displaced.

- Determining the **% by mass of $CaCO_3$** in the garden lime sample:
  Notice the relationship between CO_2 and $CaCO_3$ (Equation 1). Now consider the gas laws in the Gas Chapter of your class textbook (Boyles' Law, Charles' Law, Avogadro's Law, Ideal Gas Law, and Guy-Lussac's Law). Which one should you use to calculate the moles of carbon dioxide produced in this experiment? How will you determine the mass of $CaCO_3$ that reacted?

  The percent by mass of calcium carbonate can be calculated using Equation 3.

$$\%\,CaCO_3 = \frac{mass\ CaCO_3,\ g}{mass\ garden\ lime,\ g} \times 100 \qquad \textit{Equation 3}$$

You will perform the analysis in triplicate, and report the mean percent by mass of calcium carbonate and the standard deviation of the percent by mass of calcium carbonate (see Equation 4 and Equation 5 below).

$$\text{Mean:} \quad \bar{x} = \frac{x_1+x_2+x_3}{n} \qquad \textit{Equation 4}$$

$$\text{Standard deviation:} \ s = \sqrt{\frac{\sum(x_i-\bar{x})^2}{n-1}} \qquad \textit{Equation 5}$$

where: n is the number of trials
x_i is the individual result of each trial

In addition, you will calculate the relative percent error using Equation 6, and state if the sample is within acceptable results or not.

$$Relative\ \%\ error = \left|\frac{true\ value - calculated\ value}{true\ value}\right| \times 100\% \qquad \textit{Equation 6}$$

Example 1: Determining the percent of calcium carbonate in ground garden lime sample 37:

Sample 37 was analyzed in triplicate following the setup shown in Figure 4. The mass of the garden lime for Trial 1 was 2.195 g. After mixing it with HCl(*aq*), 325 mL of water were displaced at 23.6 °C. The atmospheric pressure (displayed by the barometer in the room) was 29.86 inches Hg. The vapor pressure of water was found by using Appendix 5. The sample was analyzed two more times. Following are the results for the percent by mass of calcium carbonate calculated for each trial:

Trial 1	**Trial 2**	**Trial 3**
59.0 %	57.8%	61.4%

The mean, the standard deviation, and the relative percent error were calculated.

Conclusion: The percent of $CaCO_3$ in sample 37 is 59.4% ± 1.8%. The relative percent error is 15.1%. The mean % $CaCO_3$ in Sample 37 is not acceptable.

(You are asked to verify these results in Prelab Question 3.)

Techniques

- Technique 1: Cleaning glassware
- Technique 2: Using a balance
- Technique 3: Transferring liquids
- Technique 4: Using a graduated cylinder
- Technique 11: Disposing chemical waste
- Technique 18: Measuring temperature

Technique

List of Chemicals

- Alka-Seltzer
- garden lime
- 3 M hydrochloric acid

List of Equipment and Glassware

- one 25-mL buret
- one 50-mL beaker
- one 250-mL beaker labeled as waste
- one 1000-mL Pyrex beaker
- one 2000-mL plastic beaker (or equivalent)
- one Florence flask
- one two-way connective tubing stopper
- one thermometer
- one 1000-mL graduated cylinder
- one small funnel
- one glass rod
- one small side-arm flask
- one watch glass

Experimental Procedure

Part A Sample Preparation and Apparatus Setup

1. **Water Saturated with CO_2.** Add about 1500 mL of tap water into a 2000-mL plastic beaker. Saturate this water with carbon dioxide by adding ¼ of an Alka-Seltzer tablet. Stir with a glass rod. Once the Alka-Seltzer has dissolved, try to remove as many gas bubbles as possible by stirring the water with the glass rod or tapping it gently.

Technique 2

2. **Sample preparation**. Obtain an unknown sample of the ground garden lime. These are located on the professor's bench (or other designated area). Record the unknown number on the Experimental Data and Calculations sheet. Measure approximately 2.3 g of the sample. Record the mass in Data Table 1. Transfer the sample into a small side-arm flask.
3. **Set-up of CO_2 collection system.**
   Set up the CO_2 generator and gas collection apparatus as shown in Figure 4:
   a. Completely fill the Florence flask with the CO_2-saturated water prepared in step A1.
   b. Place the stopper with the connective tubing ensemble in the Florence flask (you might want to do this over the sink or place paper towels under it since it will overflow). Careful that you do not move the flask vertically. If you do, you will produce a siphon (water will flow out of the tubes).

Technique 3

   c. Make sure there is no air trapped under the stopper. (It is OK if the air bubbles are small, otherwise remove the stopper, add more water, and place stopper on again.)
   d. Notice the difference in length of the two glass tubes **below** the stopper on the connective tubing ensemble. Connect the hose coming from the glass with shortest length (below the stopper) to the side-arm flask (first secure the flask to the stand with a utility clamp).
   e. Place the buret on top of the side-arm flask. Make sure the stopper is tightly in place.
   f. Place a pinch clamp through the hose coming from the glass tube with the longest length, then place this hose in a 1000-mL Pyrex beaker. Make sure the tip of the hose rests on the bottom of the beaker and the clamp rests in such a way that it does not pull the hose out of the beaker.
4. **Acid delivery system**.
   a. Obtain a 25-mL buret.
   b. Pour about 30 mL of 3 M HCl in a 50-mL beaker.
   c. Rinse the buret (use a small funnel) with three small portions of 3 M HCl, making certain that the solution wets the entire inner surface. Drain each rinse through the buret tip.
   d. Fill the buret with HCl using a small funnel. (The purpose of the buret is to deliver the HCl in a closed system; the HCl will be the excess reagent, so you do not need to monitor the volume added.)

Golden Lab Rules:

1) Label the HCl beaker.
2) Collect the rinses in the waste beaker.
3) Cover the remaining HCl with a watch glass.

   e. Remove the funnel (place on a watch glass or in a beaker; do not place it directly on the bench). Secure the buret on stand. Place it on the side-arm flask when you are ready to start the reaction. (You might find it easier to remove the utility clamp at this point.)
   f. Make sure all connections are tight. The gas generated should not escape the system.

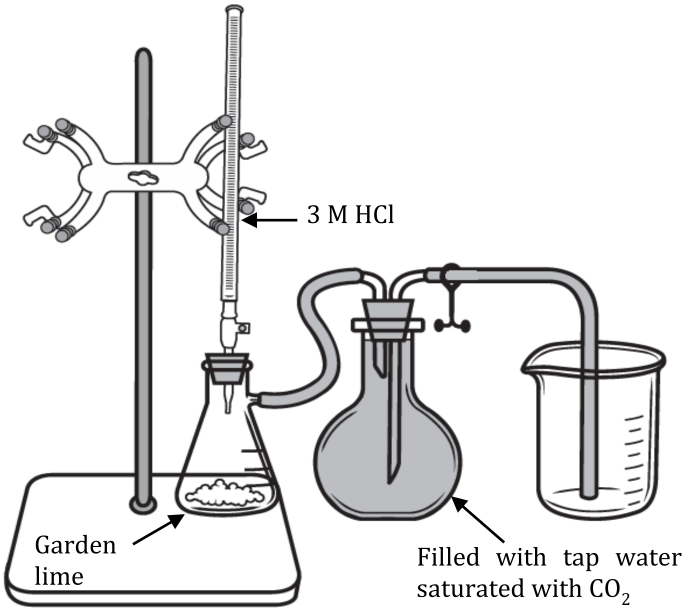


Figure 4: CO_2 Generator and Gas Collection Apparatus.

(Not to scale.)

Part B Collection of the Carbon Dioxide

1. Make sure that the **pinch clamp is open** and the hose in the 1000-mL beaker rests on the bottom of the beaker throughout the reaction.
2. Add small portions of HCl to the side-arm flask by turning the valve on the buret a complete full circle. Do not leave it open because the gas will escape through the buret. Add at least 6 mL of HCl.
3. Continue to add HCl as needed, but do not add so much that the HCl reaches the arm of the side-arm flask – you do not want the acid to go into the tubing.
4. Gently shake the side-arm flask to ensure that the acid continues to react with the calcium carbonate in the garden lime.
5. The reaction is complete when, upon addition of HCl and stirring, no more gas bubbles are produced.

Part C Measurement of the volume, temperature, and pressure of the CO_2

1. **Equilibration of the internal pressure** in the Florence flask with the room pressure:
    a. At this point, you have two containers with water: the 1000-mL beaker and the Florence flask. Which volume is lower, the one in the flask or the beaker? Physically raise the one that has the lower volume until the top layer of the water is as high as the top layer of the water in the other container. See Figure *3*.
    b. As soon as the levels are at the same height, open the pinch clamp, and place the hose between the clamp legs so that it closes (pinches) the hose. Place the container back on the tabletop.

    c. Remove the hose from the beaker.
    d. Write your signature in Data Table 2 to indicate that you completed this adjustment.
2. **Temperature**: Measure the temperature of the displaced water and record it in Data Table 2.
3. **Volume**: Pour the water displaced into a 1000-mL graduated cylinder. Read and record the volume in Data Table 2.
4. **Barometric Pressure**: Determine the room pressure using the barometer located on the professor's bench. Record the value in Data Table 3. This pressure is equivalent to the pressure of the CO_2 plus water vapor in the Florence flask because the pressures were equilibrated prior to removing the hose.
5. Remove the side-arm filtration flask and pour the contents into the Acid/Base Waste container. Rinse it well with deionized water. Refill the Florence flask with the CO_2-saturated water (you may reuse the water that is in it as well as the water that was displaced).
6. Determine if you need to refill the buret with HCl. The next Trial should take approximately the same amount as the first Trial. If there is not enough HCl, add some more.
7. Repeat above procedure for Trials 2 and 3.

Technique 18
Technique 4

Part D Calculations

1. Record the vapor pressure of water in Data Table 3, and convert to the units requested in the Table. Calculate the pressure of the CO_2 collected for each trial. Show calculations for Trial 1 under the Table.
2. Calculate the moles of CO_2 collected: discuss with your lab partner which gas law you will use for this calculation. Review the data collected. Convert data to required units.
3. Calculate the % $CaCO_3$ for each trial. Record in Data Table 4. Show calculations for Trial 1 under the Table.
4. Calculate the mean % $CaCO_3$ and its standard deviation. Record in Data Table 5.
5. Calculate the relative % error of the mean % $CaCO_3$ with reference to the expected value. Record in Data Table 5. Show calculations under the Table.

Clean up/Disposal

- When finished, pour the contents of the side-arm flask, buret, and any acid left, into your Waste beaker. Rinse them with water; collect the rinse in the Waste beaker.
- Transfer the contents of your Waster beaker into “Acid/Base Waste” container.
- Rinse the buret with water and allow it to drain through the tip. Place it, tip up, with the valve open, in the buret canister.
- Wash the side-arm flask with soap and water. Rinse with deionized water.
- Pour the CO_2-saturated water down the drain and dry the outside of the glassware. (Not necessary to wash -only exposed to CO_2-saturated water). Return all equipment and glassware to its original location.

Technique 11

Pre-lab

1. Read the Introduction and Experimental Procedure. List all the data that needs to be measured.
2. You will need to weigh approximately 2.3 g of the ground garden lime. Write a detailed procedure on how to weigh it.
3. Using the data in Example 1, perform the following calculations, <u>showing all your work</u>.
    a. Calculate the moles of carbon dioxide produced in Trial 1. *Hint:* Consider which gas law you will use for this calculation. Which measurements require unit conversion for the law? (See pages 2 - 4.)
    b. Calculate the mass of calcium carbonate in Trial 1.
    c. Verify that the % calcium carbonate in Trial 1 was calculated correctly.
    d. Verify that the mean % calcium carbonate in sample 37 was calculated correctly.
    e. Verify that the standard deviation was correctly reported.
    f. Verify that the relative percent error was correctly reported.
4. A pure sample of calcium carbonate produced 0.998 g CO_2. What was the mass of the calcium carbonate used?
5. How many mL of 3.0 M HCl are needed to completely react with 2.500 g of pure calcium carbonate? [Note: You will use less than this calculated volume of HCl for each trial. Think about the maximum volume of HCl you will need. To prevent waste, do not take more than what you need to perform the experiment.]

Post-lab

<u>In the data/calculations section:</u>

1. Include the data collected in the lab.
2. Write the balanced chemical equation that relates CO_2 and calcium carbonate.
3. For Trial 1:
    a. Show calculation for the pressure of the CO_2.
    b. Show calculation for the percent by mass of calcium carbonate.
4. Calculate (show calculations):
    a. the mean percent by mass of calcium carbonate
    b. the standard deviation
    c. the relative percent error

<u>Write a conclusion (see Appendix 9). Include:</u>

1. The unknown number.
2. The mean % mass $CaCO_3$ with its standard deviation.
3. The relative % error of the mean % mass $CaCO_3$.
4. State if the unknown garden lime that you analyzed has an acceptable percentage $CaCO_3$.

References

1. Klein, C. *Vegetable Gardening*. I-5 Publishing, LLC. UK. **2016,** 21, 30.
2. Beran, J. A. *Laboratory Manual for Principles of General Chemistry*, 6th ed. John Wiley & Sons: New York, NY, **2000**, 237-244.

VALENCIA COLLEGE

Experiment 7: Analysis of a Gaseous Product
Experimental Data and Calculations

Name: ______________________________ **Date:** ____________________

Lab Partner: ______________________________ **Section:** ____________

Unknown Sample Number __________

Table 1. Mass of Unknown Sample Used

	Trial 1	Trial 2	Trial 3
1) Mass of weighing boat ( )			
2) Mass of weighing boat + sample ( )			
3) Mass of sample ( )			

Table 2: Determination of Temperature and Volume of the Carbon Dioxide Gas Collected

	*Trial 1	Trial 2	Trial 3
1) Adjusted water level after reaction done (student's signature)			
2) Temperature of CO_2 ( )			
3) Volume of CO_2 ( )			
4) Temperature of CO_2 (K)			
5) Volume of CO_2 (L)			

* Show calculations for Trial 1 here:

Name: ____

Table 3: Determination of the Pressure of the Carbon Dioxide Gas Generated

	*Trial 1	Trial 2	Trial 3
1) Barometric Pressure (inches of Hg)			
2) Vapor Pressure of H_2O ( ) (Appendix 5)			
3) Barometric Pressure (atm)			
4) Vapor Pressure of H_2O (atm)			
5) Vapor Pressure of CO_2 (atm)			

* Show calculations for Trial 1 here:

Table 4. Percent by Mass of Calcium Carbonate in Unknown Sample Number __________

Trial #:	% Calcium Carbonate
1) Trial 1**	
2) Trial 2	
3) Trial 3	

** Show calculations of % of calcium carbonate for Trial 1 here:

Name: ____

Table 5. Results Summary

1) Mean percent by mass of calcium carbonate	
2) Standard deviation of the percent by mass of calcium carbonate	
3) Relative percent error of the mean percent by mass of calcium carbonate	

** Show calculations for mean, standard deviation and relative percent error here:

Report: Hand in all sheets of the experimental data, calculations, and write a conclusion.

Experiment 8
Calorimetry

Version 2a
Eileen Pérez, Ph. D.
Adapted from Emily Lesher, Ph.D.,[1] and Michael W. Vannatta, Ph.D. et al.[2]

The heat capacity of a Vienna sausage is determined, and related to the amount of heat needed to treat frostbite. Several ionic compounds will be tested to determine a possible heat-generating process for a heat pack, and the selected ionic compound will be studied.

Objectives

- Measure, calculate, and correctly use the terms heat, temperature, specific heat, reaction enthalpy, endothermic reaction, and exothermic reaction.
- Understand the relationship between heat and specific heat capacity.
- Understand the relationship between enthalpy and heat.
- Calculate the amount of heat needed to heat a portion of a hand, and set up a dissolution reaction that will deliver that amount heat.

Learning Outcomes

Students will be able to:

- Identify reactions as exothermic or endothermic.
- Perform calculations involving change in temperature, specific heat capacity, and heat.
- Use scientific reasoning skills (such as observing, measuring, inferring, and predicting) to problem-solve a solution for a simulated real-world situation.
- Relate ΔH and q.

Definitions

- **Calorimetry** – quantitative analysis of heat changes in a system
- **Combustion** – chemical reaction that occurs when a substance combines with oxygen; the reaction is often rapid and often produces heat and light
- **Dissolution** – the process of a solute mixing and dispersing within a solvent to form a solution
- **Endothermic reaction** – a reaction that absorbs energy in the form of heat
- **Enthalpy** – the sum of a system's internal energy; equal to heat in a chemical process if pressure is held constant and no work is done
- **Enthalpy of combustion** – heat released during combustion reaction
- **Enthalpy of reaction** – heat associated with a chemical reaction
- **Enthalpy of solution** – heat absorbed or released when a substance dissolves in a solvent
- **Exothermic reaction** – a reaction that releases energy in the form of heat
- **Heat** – a transfer of thermal energy; energy used to cause the temperature of an object to rise (has direction – flows from hot to cold)
- **Solute** – the component in least amount in a solution

- **Solution** – a homogeneous mixture of a solute(s) and solvent
- **Solvent** – the major component of a solution
- **Specific heat capacity (C_s)**– the amount of heat needed to raise the temperature of 1 g of a substance by 1°C, units of J/g· °C
- **Temperature** – measures the average velocity of particles; a physical property that determines the direction of heat flow between two bodies in contact
- **Thermochemical equation** – a balanced reaction that includes the phases of each substance and the enthalpy of the reaction
- **Thermochemistry** – the study of heat or energy flow in chemical reactions

Introduction

You work for the research and development division (R&D) of "Warm-R-Us". Your team has been entrusted to design a heat pack for maintenance workers on the Alaskan pipeline. Your heat pack must be able to treat frostbite in a worker's hand (heat should be applied gradually, not directly; the treatment process would be addressed in a future phase in the development of this product). Today your team will focus on performing preliminary tests to develop a heat pack. That is why you will do each test only once. Once your team feels comfortable with the preliminary results, the next phases involve repeating tests, and meeting with the engineering department to create a prototype. You will not be participating in these next phases.

Heat and the Human Body

The human body works best within a very narrow temperature range. A temperature drop as little as 2°C in the body's core causes hypothermia symptoms such as mental difficulties and loss of physical coordination. Much more extreme temperature loss may be survived by the extremities, but can lead to frostbite if the flesh freezes. Victims of hypothermia require immediate treatment, and in outdoor situations, the treatment is often warmth provided by portable heat sources, such as heat packs. Heat packs are available that produce warmth through various chemical reactions. Such heat packs are convenient because they only release heat when triggered. One common heat pack contains an internal pouch of water and a solid powder. Once the pouch of water is broken open, there is an exothermic reaction between the water and the powder. These heat packs have limitations. For example, they do not work well in extreme cold - the water will freeze.

In the coldest environments, heat packs are available that contain only the powder in a resealable waterproof sack. When heat is needed, the sack can be opened and any aqueous solution poured inside. The sack is resealed and the reaction produces heat. Any aqueous solution will work - melted snow, stream water, coffee, even urine. Some other heat packs do not require water – they are not based on solubility. For example, Thermacare® uses the heat produced by the oxidation of iron powder with oxygen.

As part of this lab, you will:

- measure the amount of heat involved in frostbite
- examine a possible heat-generating reaction for a heat pack
- use calorimetry to measure the heat generated by 1 gram of the reaction selected
- based on results of the heat generated by the reaction selected, calculate the amount needed to heat an average adult person's hand between 37°C and 40°C (a temperature above 40°C has the potential of burning the person treated)

Heat and Temperature

If an object (such as a pot of water) is positioned to absorb the heat given off during a chemical reaction, then the temperature of the object will change. Equation 1 allows us to determine the heat associated with the temperature change:

$$q = m \times C_s \times \Delta T \qquad \textit{Equation 1}$$

where: $q =$ the amount of heat absorbed by the object, in J
$m =$ the mass of the object being heated, in g
$C_s =$ the specific heat capacity of the object, in $J/g \cdot °C$
$\Delta T =$ the change in temperature of the object, $T_f - T_i$

The specific heat capacity is different for different substances, as can be seen in Table 1.

Table 1. Specific Heat for Selected Substances

Substance	**Specific Heat Capacity** ($J/g \cdot °C$)
water	4.18
air	1.01
aluminum	0.897
granite	0.790

Exercise 1: How much heat is needed to raise the temperature of 5.00 g of granite from 25.0 °C to 40.0 °C?

$$q = m \times C_s \times \Delta T$$
$$= (5.00\ g)\left(0.790\ \frac{J}{g \cdot °C}\right)(40.0°C - 25.0\ °C)$$
$$= (5.00\ g)\left(0.790\ \frac{J}{g \cdot °C}\right)(15.0\ °C) = 59.\underline{2}5 = 59.3\ J$$

When two objects at different temperatures are placed in contact, heat flows from the substance at the higher temperature to the substance at the lower temperature until both materials reach the same final temperature. Based on the Law of Conservation of Energy, the amount of heat energy lost by the hot material equals the amount of heat gained by the cold material. Stated mathematically:

$$q_{material\ 1} = -q_{material\ 2} \qquad \textit{Equation 2}$$

The minus states that the heat change of material 2 is opposite in sign of material 1, not that material 2 is always negative. This relationship is usually used in calorimetry.

Calorimetry

Many experiments in thermochemistry involve a calorimeter. A calorimeter is simply a container that insulates a reaction from the surrounding environment. Usually, a calorimeter will have a water bath that changes temperature; up or down depending on whether the reaction is exothermic or endothermic, or whether an object is placed in the bath that is hotter or colder than the water. For many aqueous reactions, **the water bath is the solution itself**, as in the second part of this lab. Your calorimeter (see Figure 1) will consist of a thick Styrofoam cylinder closed on one end plus a Styrofoam lid (or one plastic lid plus two Styrofoam coffee cups, one nestled inside the other). Styrofoam is an excellent insulator, but with time, heat will slowly leak out of the cup. By plotting temperature of the solution versus time (see Figure 2), you will be able to extrapolate to determine the final temperature of the reaction more precisely. You will collect the data using a LabQuest Data Collector with a temperature probe. This system will collect the data for you and graph it.

Figure 1. Calorimeter setup.

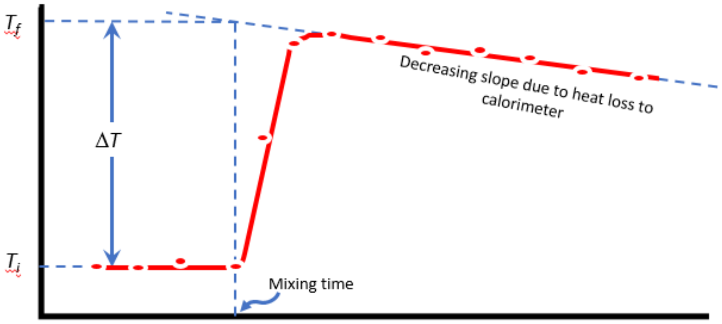


Figure 2. Temperature vs time for an exothermic reaction. (The intercept between the trendline of the decreasing slope and the vertical line at the time of mixing corresponds to the final temperature.) Image courtesy of Diego J. Diaz Lopez.

Exercise 2: A 55.0 g piece of metal was heated in boiling water to 99.8 °C and then dropped into a calorimeter containing 25.0 mL of water with an initial temperature of 23.4 °C. A graph similar to the one shown in Figure 2 was obtained. Using the graph, the final temperature of the metal and water was determined to be 26.1 °C. What is the specific heat of the metal? The density of water is 1.00 g/mL.

$$q_{H_2O} = m_{H_2O} \times C_{s\,H_2O} \times \Delta T_{H_2O} = \left[(25.0\ mL)\left(1.00\ \frac{g}{mL}\right)\right](4.18\frac{J}{g\cdot°C})(26.1\ °C - 23.4\ °C)$$

$$= (25.0\ g)\left(4.18\frac{J}{g\cdot°C}\right)(2.\underline{7}\ °C) = 2\underline{8}2.15\ J = 280\ J$$

$$q_{metal} = -q_{H_2O} = (-1)(2\underline{8}2.15\ J) = -2\underline{8}2.15 = -280\ J$$

To find the specific heat capacity of the metal use the calorimetry equation again:

$$q_{metal} = m_{metal} \times C_{s\,metal} \times \Delta T_{metal}$$

Solving for specific heat capacity, $C_{s\text{ metal}}$, of the metal and substituting values:

$$C_{s\,metal} = \frac{-2\underline{8}2.15\text{ J}}{(55.0\ g)(26.1℃ - 99.8℃)}$$

$$= \frac{-2\underline{8}2.15\text{ J}}{(55.0\ g)(-73.\underline{7}℃)} = 0.06\underline{9}606 = 0.070\ J/g \cdot ℃$$

Enthalpy of a Reaction

An important part of modern chemistry involves studying energy changes that occur during chemical reactions. These energy changes are of fundamental importance in understanding the "driving force" of a chemical reaction. The most common way energy is exchanged between a chemical system and the environment is by evolution or absorption of **heat, *q***. The change in heat energy accompanying a chemical reaction at constant pressure (q_p) is known as **enthalpy change, ΔH**. By convention, reactions in which **heat is absorbed** are labeled **endothermic** and have **positive values of ΔH** ($+q_p = +\Delta H$); reactions in which **heat is released** are labeled **exothermic** and have **negative values of ΔH** ($-\ q_p = -\ \Delta H$). Cold packs used in athletics are familiar to many sports enthusiasts. In order to derive coldness from the pack, a plastic packet of water is broken inside another packet containing a solid salt such as NH_4NO_3. In this case the **enthalpy of solution** is endothermic indicating that heat is absorbed as the salt dissolves, as can be seen in the thermochemical equation for NH_4NO_3 (see Equation 3). Thus, the enthalpy of solution is designated with a positive sign.

$$NH_4NO_3(\text{s}) \overset{H_2O}{\rightarrow} NH_4{}^{+} + NO_3{}^{-}(\text{aq}) \qquad \Delta\text{H} = +25.70\text{ kJ} \qquad \textit{Equation 3}$$

The heat absorbed by this reaction, q_{rx}, can be measured using calorimetry. In this case, since the salt dissolves in the water, the heat absorbed or lost by the solution, q_{sln}:

$$q_{sln} = m_{sln} \times C_{s_{sln}} \times \Delta T_{sln} \qquad \textit{Equation 4}$$

where: $m_{sln} = mass_{salt} + mass_{water}$

$$C_{s_{sln}} = C_{s_{H_2O}}$$

$$\Delta T_{sln} = T_{f_{sln}} - T_{i_{water}}$$

$$q_{rx} = -\ q_{sln}$$

The amount of enthalpy (ΔH) or heat (q) absorbed or released by any reaction is calculated with Equation 5.

$$\Delta H = (mol\ of\ chemical\ x) \times \frac{\Delta H_{reaction}}{coefficient\ of\ chemical\ x} \qquad \textit{Equation 5}$$

Exercise 3: Determine the enthalpy absorbed by the dissolution of 125.0 g of solid ammonium nitrate in water.

This can be solved using Equation 5 and Equation 3:

$$?\,kJ = 125.0\ g\ NH_4NO_3 \times \frac{1\ mol\ NH_4NO_3}{80.052\ g\ NH_4NO_3} \times \frac{+25.70\ kJ}{1\ mol\ NH_4NO_3} = +\,40.1\underline{3}0165 kJ$$

$$= +40.13\ kJ$$

If we do not know the enthalpy of the reaction, but we have measured the heat involved with a certain mass, we can use this relationship to find another heat-to-mass or mass-to-heat relationship for the same reaction or process. This is presented in exercise 4.

Exercise 4: When 3.005 g of solid $MgSO_4$ were dissolved in 100.0 mL of water, the temperature of the water rose from 21.0 °C to 24.3 °C. This reaction is being considered by another team as a possible source for the heat pack. The heat needed to warm the surroundings is 2238 J. How many grams of $MgSO_4$ should be dissolved in 100.0 mL of water to generate 2238 J? Assume that the density of water is 1.00 g/mL.

Since the $MgSO_4$ will dissolve in the water, use Equation 4 to determine q_{sln}:

$$q_{sln} = (100.0\ g + 3.005\ g) \times \left(4.18 \frac{J}{g{\cdot}°C}\right) \times (24.3°C - 21.0\ °C)$$

$$= (103.005\ g) \times \left(4.18 \frac{J}{g \cdot °C}\right) \times (3.3\ °C) = 1\underline{4}20.85\ J = 1.4 \times 10^3\ J$$

The heat of dissolution of 3.005 g of $MgSO_4$ in 100.0 mL of water is:

$$q_{MgSO_4} = -q_{sln} = -1\underline{4}20.85\ J$$

Therefore the heat per g of $MgSO_4$ would be:

$$q_{MgSO_4}/g = \frac{-1\underline{4}20.85\ J}{3.005 g} = -4\underline{7}2.828\ J/g$$

Now use this relationship to find the mass associated with −2238 J (remember: generates, so it is an exothermic process).

$$?\,g_{MgSO_4} = -2238\ J \times \frac{1\ g}{-4\underline{7}2.828\ J} = 4.\underline{7}33 = 4.7\ g$$

Techniques

- Technique 2: Using a balance
- Technique 4: Using a graduated cylinder
- Technique 18: Measuring temperature
- Technique 20: Using the LabQuest data collector with temperature probe

List of Chemicals

- Vienna sausage
- NaCl
- KCl
- $CaCl_2$

List of Equipment and Glassware

- one LabQuest Data Collector with a thermal probe
- one 250-mL beaker
- one 18 mm × 150 mm test tube
- one test tube rack
- one crucible tongs
- one Vienna sausage
- one small beaker or watch glass
- one calorimeter with its lid
- one magnetic stirrer
- one magnetic bar
- one 100-mL graduated cylinder or larger
- six 13 mm × 100 mm test tubes
- one ruler that measures cm
- one thermometer
- stand and utility clamp

Experimental Procedure

This experiment will require most of the time allocated to the lab period. If you are not well prepared and do not use your time wisely, you will not be able to complete this experiment. Points will be deducted for areas not completed. You will need to deal with deadlines and crunch times when you work in private industry. Preparation is key, as well as time management and proper coordination with your team member(s).

Part A Estimation of the Heat Capacity of a Human Hand

Obviously, heat must be taken from a person's hand to cool it down, and heat must be added to warm it up again. However, how much heat is governed by the hand's **specific heat capacity**. To know how much heat is needed to warm a hand, you first need to estimate the specific heat capacity of a hand. Your research team has noticed the eerie similarity between a Vienna sausage and a human hand. Once you determine the heat capacity of a Vienna sausage, you will use it to estimate the heat capacity of an average size adult human hand.

1. One team member should collect a LabQuest Data Collector with the thermal probe and instructions sheet, and proceed to set it up following the instructions sheet. **Set the "Interval" to 5 s/sample and the "Duration" 1000 s. Re-enter these settings before each new run. (**Ignore the rate; the LabQuest automatically adjusts it when the interval is entered.)

Technique 20

2. Meanwhile, the other team member should:
    a. Collect a 250-mL beaker, an 18 mm X 150 mm test tube, a test tube rack, crucible tongs, and one Vienna sausage (use the tongs to place it in a small beaker or watch glass to transport it to your area - don't squish it).
    b. Measure the mass of the Vienna sausage (do not place it directly on the balance). Record this value in Data Table 1.
    c. Create an ice slush in the 250-mL beaker: fill the beaker with ice and add just enough water to produce a slush (see Fig. 3). The goal **IS NOT** to make ice water, but to make packed ice with water between the pieces of ice.
    d. Add tap water to the test tube until it is approximately ¾ full and leave it in the test tube rack (it will be used later to return the probe to room temperature before placing it in the calorimeter).

Technique 2


Figure 3. Ice slush.

3. Skewer the Vienna sausage lengthwise with the temperature probe and plunge the Vienna sausage and temperature probe into the ice slush. If the ice slush melts into ice water during the experiment, pour off some water and refill with ice to recreate the slush. Leave it there until the sausage is at constant temperature (the LabQuest is displaying the sausage's temperature). Monitor the time – you want it to be in the ice slush for at least 12 minutes to achieve a temperature near that of the ice bath. Meanwhile, continue with the next steps.
4. Obtain a calorimeter with its lid and a magnetic bar. (If using two coffee cups, place the cups in a 250-mL beaker for stability).
5. Weigh the calorimeter with its lid.
6. Add 100.0 mL of deionized water into the calorimeter, and reweigh the calorimeter with its lid. Calculate the mass of the water.
7. Place the magnetic bar in the calorimeter.

Technique 4

8. Place the magnetic stirrer on the stand; place the calorimeter on the magnetic stirrer and turn the stirrer on so that the magnetic bar is gently stirring the water without splattering. Place the calorimeter lid on the calorimeter.
9. Place a utility clamp on the stand and position it is slightly above the **outer** hole of the lid. Your setup should look like the one shown in Figure 1 minus the temperature probe – do not remove it from the sausage yet.
10. Has it been at least 12 minutes since you placed the sausage in the ice slush? If so, record the temperature of the sausage in Data Table 1 (T_i). (The temperature is being measured by the LabQuest Data Collector.)
11. Remove the temperature probe from the sausage (but leave the sausage in the ice bath), wipe it with a paper towel and insert it into the test tube containing water to restore it to room temperature (wait until its temperature stops increasing as displayed on the LabQuest).
12. Remove the temperature probe from the test tube and dry it with a paper towel. Suspend it from the clamp and insert it through the outer hole on the lid. Adjust the clamp so that the probe is submerged as low as possible in the water but high enough above the lid that the lid can rotate. Make sure that the stirrer is not hitting the probe.

Golden Lab Rule: *Be careful not to poke a hole in the calorimeter with the probe.*

13. Wait until the temperature probe equilibrates with the temperature of the water. Then press play on the LabQuest Data Collector to begin data collection. After obtaining three or four readings (after 15-20 seconds) at the same temperature, rotate the lid and quickly transfer the sausage from the ice slush into the calorimeter. To transfer, pick up the Vienna sausage with crucible tongs, and quickly shake any ice pieces and excess water from the sausage before putting it into the calorimeter. Be careful not to splash any water out when you put the sausage in the calorimeter. Replace the lid. (While the data is being collected, one of the team members should start collecting the chemicals stated in steps B1 and B2.)
14. Stop data collection once the temperature starts increasing at a steady pace. Save the run.
15. Dispose of the Vienna sausage in the regular trash can. Pour the water in the calorimeter down the drain and invert the calorimeter over a paper towel. Meanwhile, the other team member should print the graph (print one copy for each team member) following the instructions sheet located next to the laptops.
16. Using a pencil, draw two lines on the graph as shown in Figure 2:
    - draw a vertical line parallel to the y-axis at the mixing point
    - draw a trendline for the data collected in the region of increasing/decreasing slope due to heat gain/loss of the water; extend it until it intersects the mixing line

    The "y" value of this intersection point corresponds to T_f. Record the initial and final temperatures of the water (T_i and T_f) in Data Table 1.
17. Complete Data Table 1, including calculations.
18. The average size of an adult person's hand is approximately equivalent to 16 Vienna sausages. Based on the mass of the Vienna sausage you weighed, estimate the mass of an average adult person's hand. Then calculate how much heat would be required to raise the temperature of an average adult person's hand from 15.0 °C to 37.0 °C. Record this value in Data Table 2.

Part B Selecting a Material for the Heat Pack

There are three options for salts to use in the heat pack: NaCl, KCl, and $CaCl_2$. In this part of the procedure, you will do a quick screening to determine which produces heat when it dissolves in water. Record this data in Data Table 3**.**

The dissolution reaction for the first salt is shown in Equation 6.

$$NaCl(s) \overset{H_2O}{\rightarrow} Na^+(aq) + Cl^-(aq) \qquad \textit{Equation 6}$$

1. Obtain six 13 mm × 100 mm test tubes. Add approximately 2 cm of deionized water to three of these test tubes (rulers are available in the lab).
2. Add approximately 1 cm of NaCl into the 4th test tube, approximately 1 cm of KCl into the 5th test tube, and approximately 1 cm of $CaCl_2$ into the 6th test tube (label each test tube).
3. Using a thermometer, measure and record the temperature of the water in one of the test tubes.
4. Remove the thermometer and pour this water into the test tube containing the NaCl. Place the thermometer inside of this mixture and stir gently with the thermometer. Be extra careful to prevent breaking the thermometer.

Technique 18

5. Record the temperature of this solution after 30 seconds.
6. Rinse the thermometer with water, wipe it dry and place it in another test tube containing water.
7. Repeat Steps 3-6, but use KCl in place of NaCl.
8. Repeat Steps 3-6, but use $CaCl_2$ in place of NaCl.
9. Below table 3, write a note on which salt would work best to fashion a heat pack, and why.

Golden Lab Rule: *Collect rinsed chemicals in waste beaker.*

Part C Measure Enthalpy per Gram Produced by Dissolution of Selected Salt

Record the data collected from this part in Data Table 4 (create this table).

Technique 4

1. Using a paper towel, dry the calorimeter and lid previously used.
2. Add 75.0 mL of deionized water to the calorimeter.
3. Measure the mass of the calorimeter, lid and water. Calculate the mass of the water.
4. Add a magnetic bar into the calorimeter.
5. Place the calorimeter on the magnetic stirrer and place the temperature probe through the outer hole on the lid. Turn on the magnetic stirrer. Again, make sure the water does not splatter, the magnetic bar is not hitting the probe, and the probe is submerged in the water as much as possible while at the same time allowing enough space so that the probe does not touch the bottom of the calorimeter and the lid can rotate.
6. Weigh and record the mass of a boat.
7. Add approximately 1.0 g of the selected salt into the boat. Record the mass of the boat and salt.
8. After re-entering the "Interval" and the "Duration" stated in Part A on the LabQuest Data Collector, press play to begin data collection. After obtaining three or four readings, rotate the lid, and quickly transfer the measured salt into the calorimeter. If some of the salt adhered to the boat, reweigh it. Use this value as the initial mass of the boat so that you can calculate the exact mass of salt transferred into the calorimeter.
9. Stop data collection once the temperature starts decreasing or increasing at a steady pace. Save the file.
10. Print the graph (one for every team member), add a title, and draw the two lines as done previously. Record the initial temperature of the water, T_i, and the final temperature of the solution, T_f.
11. Calculate ΔT_{sln}, the heat change of the solution and the enthalpy of the reaction per gram of salt. In your calculations, you can assume the following:
    - the mass of the solution is the mass of the salt plus the mass of the water
    - the specific heat of the solution is the same as that of water, 4.18 J/ g·°C

Part D Completing the Heat Pack Design

As stated previously, your goal is to design a heat pack for maintenance workers on the Alaskan pipeline. Your heat pack must be able to treat frostbite in a worker's hand without burning them.

Calculate the mass of the salt selected that is expected to deliver the amount of heat needed to treat an average adult person's hand within a safe temperature range (see Table 2). Include this calculation in your report and the mass in your conclusion.

Unfortunately, you have been reassigned to a different project next week, so you will not be able to participate in the next phase of this project, which consists of performing tests to determine if the calculated mass actually heats the sausages to the desired temperature range.

Clean up/Disposal

- Dispose of the Vienna sausage in the regular trash can.
- Pour the salt solution (from the test tubes and calorimeter) into the Inorganic Waste container.
- Wash and dry the outside of all glassware and return to original location.
- Wipe the temperature probe clean.
- Delete your graphs from the LabQuest. Turn it off and remove the temperature probe. Store it as originally found in its original location. Return LabQuest directions.

Technique 11

Pre-lab

1. Read the procedure several times. You need to be extra prepared to ensure you finish this experiment in its entirety. Write a statement to this fact. If you do not feel prepared, write questions and make sure you get them clarified before coming to lab (email questions to your professor – make sure you give them enough time to reply).
2. Design Data Table 4.
3. Nitroglycerin, $C_3H_5N_3O_9$, is an unstable compound that decomposes rapidly upon ignition or impact. Below is the thermochemical equation for nitroglycerin:

$$4\, C_3H_5N_3O_9(l) \rightarrow 12\, CO_2(g) + 10\, H_2O(g) + 6\, N_2(g) + 13\, O_2(g)\ \Delta H_{rx} = -5678\ kJ$$

   What mass of nitroglycerin is needed to generate $-7285\ kJ$?

4. Propane, C_3H_8, is used by many campers to cook on the trail. The thermochemical equation for the combustion of propane is shown below.

$$C_3H_8(g) + 5\, O_2(g) \rightarrow 3CO_2(g) + 4\, H_2O(g) \quad \Delta H_{rx} = -2043\ kJ$$

   If 5.00 g of propane is burned and all the heat from this combustion is absorbed by 400.00 g of water at an initial temperature of 20.0 °C (ignore the pot holding the water), what is the final temperature of the water?

5. Write the dissolution reactions of KCl(s) and $CaCl_2$(s) in water.

Post-lab

1. Hand in all of your tables, graphs, and the calculations.
2. Write a conclusion (see Appendix 9) that includes the results associated with the goals of each part of the experiment (two goals for Part A, one for Part B, one for Part C, and one for Part D).

References

1. Lesher, E. St. Joseph's College of Maine, Standish, ME. Unpublished work, 2016.
2. Vannatta, M. W.; Richards-Babb, M.; Sweeney, R. J. Thermochemistry to the Rescue: A Novel, Calorimetry Experiment for General Chemistry. *J. Chem. Educ.* 2010, *87*, 1222-1224.

VALENCIA COLLEGE

Experiment 8: Calorimetry
Experimental Data and Calculations

Name: ______________________ **Date:** ______________

Lab Partner: ______________________ **Section:** ________

Table 1. Determination of the Specific Heat Capacity of One Vienna Sausage

1) Mass of the Vienna sausage	
2) Mass of calorimeter and lid	
3) Mass of calorimeter and lid + water	
4) Mass of the water	
5) Ti, initial temperature of water	
6) Tf, final temperature of water	
7) ΔT, change in temp of the water	
8) Heat change of water	
9) Heat change of sausage	
10) Ti, initial temperature of sausage	
11) Tf, final temperature of sausage	
12) ΔT, change in temp of the sausage	
13) Specific heat, Cs, of one sausage	

Calculations:

Name: ______________________________

Table 2. Estimation of the Heat Needed to Warm an Average Size Adult Person’s Hand

1) Estimated mass of 16 Vienna sausages	
2) Heat needed to warm 16 Vienna sausages from 15.0 °C to 37.0 °C	

Calculations:

Table 3. ______________________________

	NaCl	KCl	$CaCl_2$
1) temperature water			
2) temperature of mixture at 30 s			
3) ΔT			
4) visible observations of mixture			
5) type of reaction			

Conclusion:

Name: ____

Table _____. ____

(**Read over Part C**. Develop your own Table before coming to Lab. Note: you do not need to use all the columns and rows in the table below. Extra provided to not limit your creativity.)

$\Delta H_{rx}/mass_{salt}$ (J/g)			
Mass of salt needed to warm an adult person's average hand (Table 2)			

Calculations:

Report: Hand in Tables 1-4, calculations, and write a conclusion.

VALENCIACOLLEGE

Experiment 10
Dye Concentration Using a UV-Vis Spectrophotometer

version 2a

Lynta Thomas, Ph.D. and Laura B. Sessions, Ph.D.

In this experiment, you will determine the concentration of Allura Red Dye (FD&C Red No. 40) in a drink. By mixing a series of known concentration solutions of the dye and measuring absorbance ultraviolet-visible (UV-Vis) light for each, a calibration curve can be made to determine the unknown drink concentration.

Objectives

- Evaluate and apply the concept of solution concentration.
- Perform dilutions.
- Operate an ultraviolet-visible (UV-Vis) spectrophotometer.
- Express and interpret graphical data.

Learning Outcomes

- Understand and apply chemical quantities related to chemical compounds.
- Understand the relevance of quantum mechanics theory to the fundamental nature of light and spectroscopic data.
- Master essential laboratory techniques critical in the application of laboratory science study.

Definitions

- **Absorbance –** the amount of radiation absorbed by molecules in a sample, and therefore not transmitted through the sample, at a specific wavelength

- **Absorption spectrum –** a recording of electromagnetic radiation that is absorbed by a sample; for UV-Vis, units are typically absorbance versus wavelength in nm; the plural is spectra

- **Aliquot –** a small portion of sample solution

- **Calibration curve –** the graphic relationship of concentration to absorbance for a series of standard samples of known concentration that is used for determining the concentration of a substance in an unknown sample by comparison; it is often a straight line

- **Cuvette –** an optically transparent cell that holds the sample under study

- **Dilute –** to make a solution of lower concentration; add solvent

- **Frequency –** cycles per unit time; for example, wave crests per second in electromagnetic energy

- **Molar absorptivity –** a measure of how strongly a sample absorbs light at a given wavelength; it is a physical property of a compound
- **Organic molecule –** a molecule that contains carbon
- **Solute** – the component in lesser amount in a solution
- **Solvent** – the major component of a solution
- **Spectrophotometer –** an instrument for measuring the intensity of light transmitted, absorbed, or emitted from a sample
- **Standard solution –** a solution of precisely known concentration used to determine the concentrations of unknown solutions
- **Stock solution –** a solution of known concentration used to prepare more dilute solutions
- **UV-Vis –** an abbreviation for ultraviolet and visible
- **Wavelength –** the distance of one wave; for example, crest-to-crest length
- **Wavelength of maximum absorbance –** λ_{max} (read as lambda max), the highest point of absorbance in an absorbance band (peak); used to describe the UV-Vis activity of a sample

Techniques

The following techniques are used in the experimental procedure:

- Technique 1: Cleaning glassware
- Technique 6: Using a volumetric flask
- Technique 7: Using a graduated pipet
- Technique 11: Disposing chemical waste
- Technique 21a(Lake Nona) or Technique 21b(West): Using UV-Vis spectrophotometer

Introduction

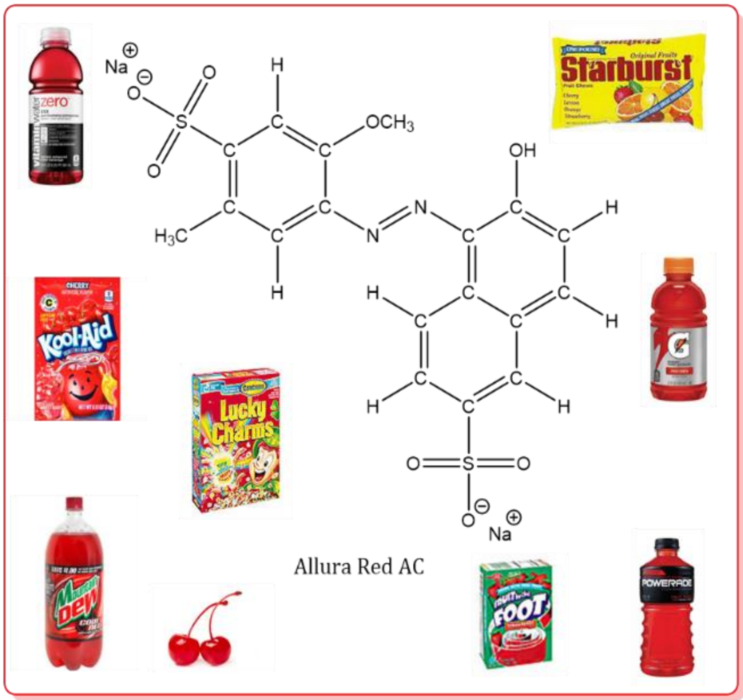


Figure 1. Chemical Structure of Allura Red AC, and Some Example Products.

Currently, there are eight synthetic food dyes approved for use in the United States by the Food and Drug Administration (FDA).[1] There has been much speculation that these artificial dyes are bad for health.[2] However, the evidence remains unclear; for example, the European Food Safety Authority and the FDA approve different dyes based upon their interpretations of the scientific research.[3] Some concern is justified given history. Since ancient times, colorants have been used to make food appear more attractive; such colorants included chalk to make bread appear more white, and lead(II) oxide and mercury(II) sulfide – toxic and poisonous heavy metal salts – to enhance the color of cayenne and curry powder and jams and candy. Allura Red AC (FD&C Red No. 40[a]) (Figure 1) is an organic molecule, a molecule that contains carbon, with chemical formula $C_{18}H_{14}N_2Na_2O_8S_2$ and molecular mass 496.42 g/mol.

In your scenario for this experiment, you have been contracted by a health organization, Don't Be Dyeing, that would like to begin research into the health effects of food dyes. They have asked you to determine the amount of Allura Red Dye in a common drink. From this amount, you can determine how many servings a consumer can drink without exceeding the FDA's acceptable daily intake (ADI) of 7.0 mg per kg of bodyweight per day.[4]

Unknown concentrations of solutions such as the amount of dye in a drink can be determined by spectroscopy, the measurement of how a substance interacts with electromagnetic radiation. Electromagnetic radiation is energy that travels through space with the characteristics of a wave. Light is specifically the visible region of the electromagnetic spectrum with wavelengths between about 400 nm and 700 nm, which is only a small portion of the entire spectrum. Our eyes detect different wavelengths of visible light as different colors. For example, light with a wavelength of 425 nm appears violet while light with a wavelength of 700 nm appears red. White light is a mixture of all the visible wavelengths.

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[a] FD&C refers to the U. S. Federal Food, Drug, and Cosmetic Act, first passed in 1938.

When an object absorbs some wavelengths of white light and reflects others, it appears colored; the observed color is predominantly the colors reflected. So, blue food dye absorbs wavelengths around 600 nm (the orange wavelengths), reflecting the blue wavelengths back to the eye (Figure 2).

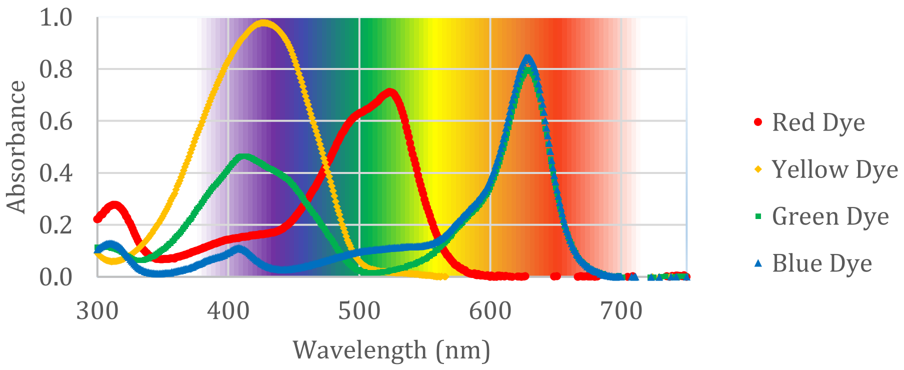

Figure 2. Ultraviolet-Visible Spectra of Food Dyes.

A color wheel can be useful for determining the color that a solution will appear based on the wavelengths absorbed (Figure 3).[5] If a solution absorbs wavelengths of one particular color, it will have the appearance of the color directly opposite it on the wheel. For example, FD&C Red No. 40 is known to absorb light around 504 nm, in the green range, therefore the solution will look red. FD&C Green No. 3 is known to absorb light around 628 nm, in the red range, therefore the solution will look green. However, mixtures of dyes can also be used. Green food dye is composed of FD&C Yellow No. 5 and FD&C Blue No. 1, and therefore absorbs wavelengths around 414 nm and 625 nm, transmitting blue and yellow together to make green.

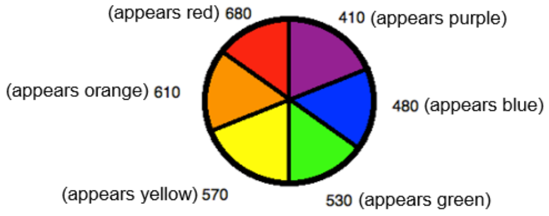

Figure 3. Color Wheel. Image used under a Creative Commons Attribution License (by 4.0) from OpenStax, Chemistry.[5]

The Beer-Lambert Law states that the absorbance of a substance is directly related to the concentration of the substance in solution:

$$A = \epsilon \cdot \ell \cdot c$$

where A is the measured absorbance, ℓ is the path length of light through the cell in centimeters, c is the molar concentration of the sample, and ϵ is the molar absorptivity or molar extinction coefficient of the sample in $cm^{-1} \cdot M^{-1}$.

The direct relationship of absorbance to concentration can be expressed by the graph of a line, and the equation of the line used to determine an unknown concentration from experimentally-determined absorbance. Let's examine how this experiment will work.

The substance in solution is placed into a cuvette, an optically transparent cell that holds the sample under study. The cuvette with its width (ℓ) is placed in the spectrophotometer along the path of the light beam (Figure 4). If a larger cuvette was used, the path length (ℓ) would increase, and more light would be absorbed. You will use one cuvette consistently to eliminate this variable.

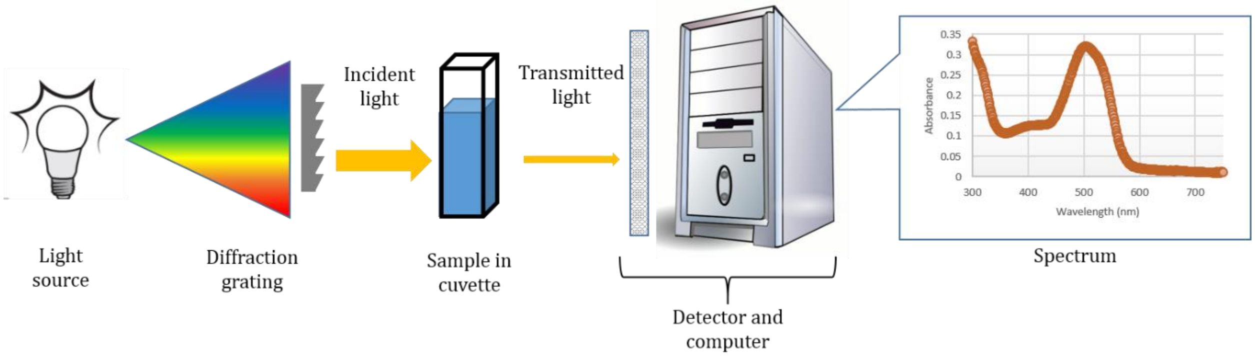

Figure 4. Simple Diagram of a Spectrophotometer.[67]

Incident light is the light that falls on a material. When light passes through the solution in a cuvette, the intensity of the incident light decreases due to absorption. The transmitted light is split into its different wavelengths by a diffraction grating and measured by the detector. If the intensity of the incident light is represented by I_o and the intensity of the transmitted light is represented by I, transmittance (T) is defined as the ratio:

$$T = \frac{I}{I_o}$$

Light absorption can also be represented as absorbance (A). Absorbance is the negative logarithm of the transmittance:

$$A = -log\left(\frac{I}{I_o}\right)$$

Molar absorptivity (ϵ) is a measure of how strongly the sample absorbs the light at a specific wavelength; it is a physical property of a compound. When working in concentration units of molarity, the unit of ϵ is $M^{-1}{\cdot}cm^{-1}$. So, absorbance is unitless. Examining the absorbance of Allura Red over the visible spectrum, you can see that the highest absorbance occurs at 504 nm (Figure 5). This wavelength is called the absorbance maximum or λ_{max}, and is read as lambda max.

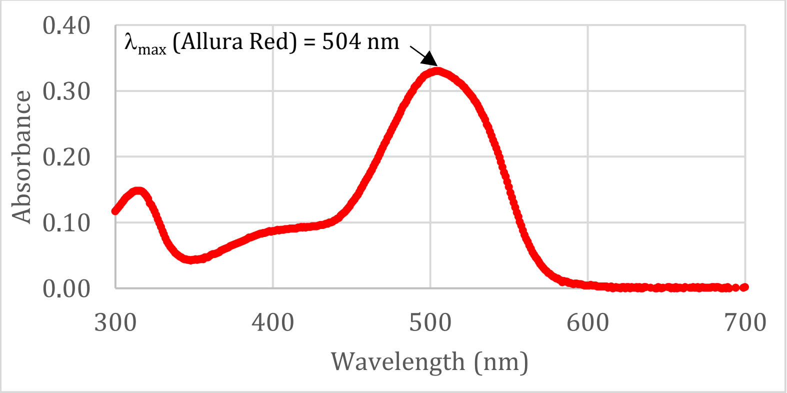

Figure 5. UV-Visible Spectrum of Allura Red AC.

On the spectrophotometer in the lab, you may be able to measure absorbance at only one wavelength at a time, so you will measure the absorbance at λ_{max}. The path length (typically 1 cm (ℓ)) and the molar absorptivity (Allura Red AC with its known ϵ) will be kept constant. By graphing the absorbance versus concentration of several known solutions, a linear calibration curve can be made (Figure 6); an unknown concentration can be determined from the equation of the line. As the amount of solute increases (with increasing concentration c per the Beer-Lambert Law), more light will be absorbed.

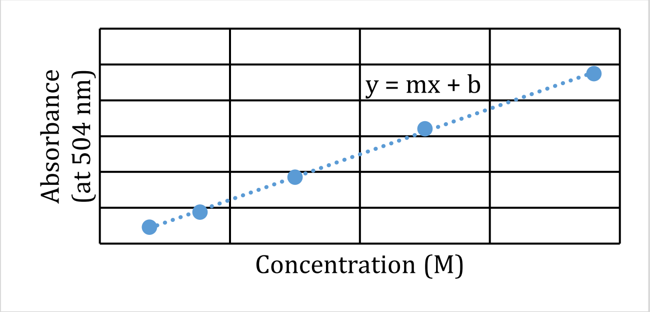

Figure 6. Beer-Lambert Graph Example.

In this experiment, you will use molarity to calculate concentrations:

$$Molarity\ (M) = \frac{moles\ of\ solute}{volume\ of\ solution\ (L)} = \frac{n}{V}$$

Solutions are stored often as concentrated stock solutions. You will require solutions less concentrated than the stock solution since the Beer-Lambert Law only works for small values of absorbance. To dilute a solution (make a solution of lower concentration) from the stock solution, a portion of the stock solution is measured into a new flask and more solvent is added. The following equation can be used to determine the aliquot, the volume of concentrated solution, needed to carry out the dilution:

$$M_1 \cdot V_1 = M_2 \cdot V_2$$

The moles of solute in the aliquot will be equal to the moles of the solute in the less concentrated or dilute solution. It is only the volume of solution that changes after dilution, hence molarity changes.

$$moles\ of\ solute\ in\ the\ aliquot = moles\ of\ solute\ in\ diluted\ solution$$

This process must be done quantitatively, which means the measurements of the quantities must be done accurately and precisely. For example, suppose an experiment requires 100.00 mL of 0.02500 M NaCl solution. The concentration of NaCl solution you have is 0.1000 M. What volume of the concentrated solution (0.1000 M) would be required for diluting to the required concentration and volume?

Upon rearranging the dilution equation:

$$V_1 = \frac{M_2 \cdot V_2}{M_1}$$

$$V_1 = \frac{(0.02500\ M\ \times 100.00\ mL)}{0.1000\ M} = 25.00\ mL$$

To prepare the 0.0250 M solution, pipet a 25.00 mL aliquot of the 0.1000 M NaCl aqueous solution using a graduated pipet and transfer the solution into a 100.00 mL volumetric flask. Dilute with deionized water to the mark with mixing as in Technique 6: Solutions Using a Volumetric Flask.

The absorbance of a series of Allura Red Dye standard solutions with known concentrations will be measured. These data will be used to make a calibration curve (absorbance (A) at λ_{max} on the y-axis and concentration on the x-axis). By comparing the absorbance of the unknown drink to the known solutions, its concentration will be determined.

Experimental Procedure

List of Chemicals

- $\sim 7.0 \times 10^{-5}$ M Allura Red AC (FD&C Red 40)
- red-colored sports drink or juice containing Allura Red
- deionized (DI) water

Since these are food products, they are non-hazardous; however, since they are in the lab, they should never be consumed. Use Safety Data Sheet (SDSs) to learn about proper handling of these chemicals. (The SDSs are available in the laboratory or online.)

List of Equipment and Glassware

- 100-mL beaker
- 50-mL beaker
- 1-mL and 5-mL graduated pipets and bulb
- funnel
- transfer pipet
- five 10-mL volumetric flasks
- UV-Vis spectrophotometer
- two cuvettes
- beaker for waste

Part A: Preparing Solutions

1. Record the exact concentration of Allura Red stock solution provided in the lab. Its concentration will correspond to M_1.
2. Pour about 30 mL of the Allura Red stock solution into a 100-mL beaker.
3. Prepare five 10-mL volumetric flasks with labels and the solutions per your pre-lab calculations. Using Technique 7 Using a Graduated Pipet and Technique 6 Using a Volumetric Flask, pipet the desired amount of stock solution into each volumetric flask, and add DI water to fill the volume to the calibration mark. Remember if the volume goes over the mark, discard the solution, rinse the flask with DI water, and remake.

Technique 6
Technique 7

Part B: Measuring UV-Vis Absorbances for the Calibration Curve

1. The instructions on calibration and use of the spectrophotometer will be found next to the instrument in the laboratory (Technique 21a (Lake Nona) or Technique 21b (West): Using a UV-Vis spectrophotometer).
2. Follow the directions to set the wavelength to 504 nm, the wavelength of maximum absorbance for Allura Red.
3. Fill the first cuvette to three-fourths its height with DI water for the blank solution. A blank solution is a solution that should contain everything (all reagents used)

Technique 21a
Technique 21b

except the substance being analyzed. We will use water as the blank since the other species in the drink should not absorb in the visible range (e.g., sugar in water is colorless and clear). "Zero out" the spectrophotometer to give only the absorbance of the sample under study.

4. Fill the other cuvette to three-fourths height with the most dilute solution.
5. Measure and record the absorbance at 504 nm.
6. Then, use the next most concentrated solutions to condition the cuvette. Fill it to three-fourths height, and measure and record the absorbance.
7. Repeat step 5 with all the solutions.
8. If any of the absorbances are greater than 1.0, check with your instructor to prepare a more dilute sample.

Part C: Measuring the Absorbance for the Unknown Drink

1. Prepare a solution of the unknown drink by diluting 1.00 mL measured with a graduated pipet into a 10-mL volumetric flask.
2. Measure and record the absorbance at 504 nm.
3. If the absorbance is greater than 1.0, check with your instructor to prepare a more dilute sample.

Clean up/Disposal

- Pour all waste into the waste container (Technique 11: Disposing Chemical Waste).
- Clean the glassware with soap and tap water, and discard in the sink. Rinse it twice with distilled water, dry the outside of the glassware, and replace in its original location (Technique 1: Cleaning Glassware).
- Place the graduated pipets tip up in the pipet canister.

Technique 1
Technique 11

Pre-lab

The pre-lab assignment must be completed before you come to the lab.

1. You will need to have the following calculations prepared for the lab. Calculate how many mL of the 7.0×10^{-5} M Allura Red stock solution will be needed to prepare 10.00 mL of the following solutions. Show your work for one example.

   3.5×10^{-5} M

   2.8×10^{-5} M

   2.1×10^{-5} M

   1.4×10^{-5} M

   3.5×10^{-6} M

2. Given the following data set (top of the next page), graph the data and determine the equation of the line. Calculate the concentration of a solution with absorbance of 0.75. Include your Excel graph, and show your calculations. Make sure to add a title and axes labels and units to the graph.

Concentration (M)	**Absorbance**
1.0×10^{-5}	0.98
8.2×10^{-6}	0.79
6.2×10^{-6}	0.59
4.9×10^{-6}	0.43
2.2×10^{-6}	0.21

3. Sudan 1 was a food dye approved by the FDA in 1918, but removed after six months for causing dermatitis;[3] now, it is listed as a carcinogen.[8] In solution, it has λ_{max} = 418 and 476 nm. What color does the Sudan 1 solution appear? Briefly explain.
4. Explain how to use a graduated pipet. Why was it chosen instead of a volumetric pipet in this experiment?
5. Print the data tables before coming to lab.

Post-lab

Show all work neatly and answer all questions under the section in complete sentences for full credit.

1. Provide the equation of the line for your calibration curve for Allura Red Dye. Plug in your absorbance value for the unknown concentration of drink to calculate its concentration in the prepared sample. Then, remember that you diluted it by ten or another factor (look back in your data table). Calculate the actual concentration of Allura Red Dye in the drink.
2. Calculate the amount (in mg) of Allura Red Dye that a consumer would obtain from an 8-ounce serving of the drink that you studied. The molar mass of Allura Red Dye is 496.42 g/mol. In one U.S. fluid ounce, there are 29.6 mL.
3. The FDA's acceptable daily intake for Allura Red Dye is 7.0 mg/kg bodyweight per day. Calculate how many servings a 200-lb person may have per day (or if you would like to, calculate how many servings you may have per day). In 1 kg, there are 2.20 lbs.
4. How many servings can a 5-year-old child, weighing 55-lb, drink in a day to remain below an acceptable daily intake of 7.0 mg/kg bodyweight?
5. Comment on any unusual or notable results. For example, if you had any extraneous data points, where might the error have occurred?
6. Include your graph at the back of the report.
7. Write a conclusion (see Appendix 9 How to Write a Scientific Conclusion).

Experiment 10: Dye Concentration Using UV-Vis Data Tables

Experimental Data and Calculations

Name: ____ **Date:** ______________________

Lab Partner: ____ **Section:** ____________

All measurements and calculated values must be reported with proper significant numbers and units. You can write any observations or description while doing the experiment.

Part A and B: Preparing Solutions and Measuring UV-Vis Absorbances for the Calibration Curve

Concentration of Allura Red AC Stock Solution ______________

Data Table 1. Preparation and Absorbance of Allura Red AC Standards.

Standard #	Volume of Stock Solution Used ( )	Concentration of Standards ( )	Absorbance
1			
2			
3			
4			
5			

Show your work for calculation of the concentration of standard 1:

Part C: Measuring the Absorbance for the Unknown Drink

Drink Analyzed: ________________________

Data Table 2. Preparation and Absorbance of Unknown Drink.

Preparation:	Absorbance
1.00 mL unknown drink diluted with DI water to 10.00 mL	

Show your work for calculation of the concentration of Allura Red AC in the Unknown Drink:

After you collect your data, prepare a calibration curve with Excel. You can follow the tutorial in Appendix 7 Using Excel to plot the graph. Plot absorbance versus concentration of the known samples in a scatter (XY) plot. Draw the "best fit line" through the data points. Use the equation for the line to calculate the unknown concentration of Allura Red in the drink as described in the Post-lab.

References

1. Color Additive Status List. U.S. Department of Health and Human Services: U.S. Food and Drug Administration. https://www.fda.gov/ForIndustry/ColorAdditives/ColorAdditiveInventories/ucm106626.htm (accessed June 24, 2017).

2. Potera, C. Diet and Nutrition: The Artificial Food Dye Blues. *Environ. Health Perspect.* **2010**, 118 (10), A428. https://www.ncbi.nlm.nih.gov/pmc/articles/PMC2957945/ (accessed June 24, 2017).

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4. Food and Drug Administration. Background Document for the Food Advisory Committee: Certified Color Additives in Food and Possible Association with Attention Deficit Hyperactivity Disorder in Children, March 30-31, 2011. http://www.fda.gov/downloads/AdvisoryCommittees/CommitteesMeetingMaterials/FoodAdvisoryCommittee/UCM248549.pdf (accessed June 24, 2017).

5. Image used under a Creative Commons Attribution License (by 4.0) from OpenStax, Chemistry. OpenStax CNX. (Accessed July 16, 2017). https://chem.libretexts.org/Core/Inorganic_Chemistry/Crystal_Field_Theory/Colors_of_Coordination_Complexes.

6. Spectroscopy Lab Experiments: Quantification of Food Dyes in Sports Drinks. http://www.stellarnet.us/spectroscopy-lab-experiments-quantification-food-dyes-sports-drinks/. (Accessed July 28, 2017).

7. Computer image from: Server-pc-workstation-computer-23312. By Clker-Free-Vector-Images. https://pixabay.com/en/server-pc-workstation-computer-23312/. (Accessed July 5, 2017).

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VALENCIA COLLEGE

Experiment 11
Molecular Modeling

version 3

Laura B. Sessions, Ph.D.

Now that we understand atomic structure, bonding, and a little about reactions, finally we come to molecular structure. Two models of aspirin are shown below: a perspective structural formula and a ball-and-stick representation (Figure 1). Models are useful for representing the three-dimensional shape and geometry of molecules, which will be important for predicting stability and reactivity in future chemistry endeavors.

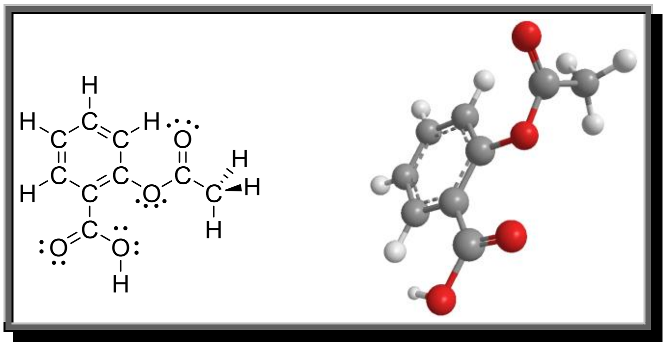


Figure 1. Molecular Model Representations of Aspirin, $C_9H_8O_4$. In the ball-and-stick model (on the right), grey indicates carbon, white indicates hydrogen, and red indicates oxygen.

Objectives

- Draw possible Lewis structures, calculate formal charges, and predict the most likely structure for a given molecular formula.
- Recognize and draw resonance structures.
- Apply the VSEPR model to predict molecular geometry.
- Relate molecular symmetry to its dipole moment and predict polarity of a molecule based on geometry and overall dipole moment.

Learning Outcomes

- Understand the nature and characteristics of a chemical bond.
- Understand the nature of molecular geometry as it relates to physical and chemical properties of molecules.

Definitions

- **Bond angle** – the angle formed by three atoms joined by bonds.
- **Electron group** – a negative region of space occupied by a group of electrons; the group can be a lone pair, single bond, double bond, or triple bond.
- **Electronegativity** – the ability of an atom to attract electrons in a bond.

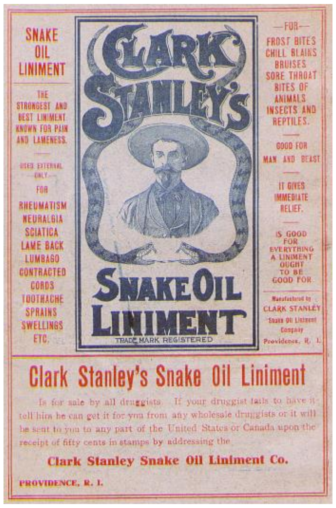
- **Electron-group geometry** – the arrangement of *electrons* around a central atom in a molecule or ion.
- **Formal charge** – the charge that an atom would have if all atoms in the molecule had the same electronegativity. While the formal charge is only a bookkeeping method for tracking valence electrons on an atom, it creates a representation of electron sharing in a molecule. The best Lewis structure will have small or zero formal charges.
- **Ionic bond** – an electrostatic attraction between ions of opposite charge. Often, ions are formed from transfer of electrons from metals to non-metals.
- **Lewis structures** – illustrate valence electrons surrounding an atom and how bonding occurs between atoms in a molecule.
- **Lone pairs** (or **nonbonding pairs**) – electrons around an atom that are not shared.
- **Molecular geometry** – the arrangement of *atoms* around a central atom in a molecule or ion.
- **Nonpolar bond** – a covalent bond between two atoms of similar electronegativity so that bonding electrons are shared relatively evenly.
- **Octet rule** – states that an atom will give up, accept, or share valence electrons to achieve a full valence shell.
- **Polar bond** – a covalent bond between two atoms with a large electronegativity difference so that electrons are held more tightly by one atom of the bond. This electronegativity difference creates a dipole, a partial negative charge area and a partial positive charge area on the molecule.
- **Resonance structures** – valid Lewis structures that differ in the placement of electrons for a molecule.
- **Valence electrons** – those in the outermost shell of an atom for main group elements, and those in the outermost shell plus the outermost *d* orbital electrons for transition metals.

Techniques

- Computer software modeling
- Ball-and-stick modeling

Introduction

John Ellis Water Machines (http://johnellis.com/) makes many promises to improve your health: using their water filtration system will 'increase the bond angle in water from 104° to 114°, which will increase blood flow, grow back hair on bald heads, and prevent Ebola'.[1] Much like Clark Stanley's Snake Oil Liniment, when claims sound too good to be true, they probably are! There are many websites, social media connections, and even brick-and-mortar stores that will take your money in exchange for unproven claims to improve health. Snake oil peddlers have been around for centuries; how can you protect yourself and your money from them? Being well-educated is one way. General chemistry can teach us about bond angles and why they occur. By the end of this lab, you should be able to predict the real bond angle of water.



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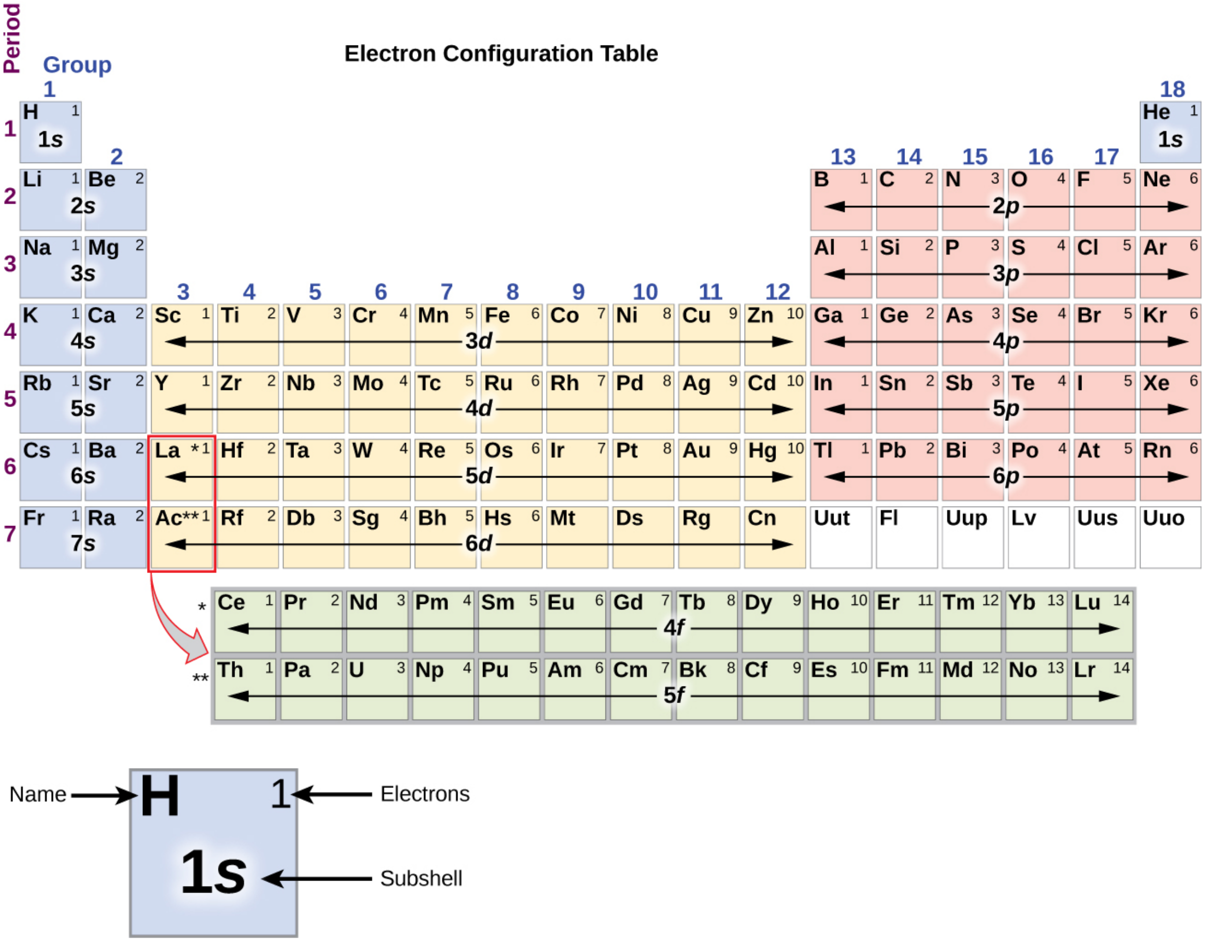
Molecules are held together by covalent bonds created by sharing electrons between two atoms. First, we start building molecules by identifying the electrons. Only electrons in valence orbitals can be shared to form bonds. We can determine valence electrons from the periodic table. Main group elements have valence electrons in the outermost shell while transition metals include the outermost shell electrons plus the outermost *d* electrons. Recall that elements in a group will have the same number of valence electrons, leading to similar chemical properties (Figure 2).



Electron Configuration Table

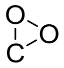
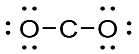
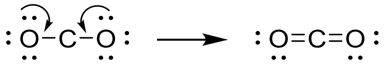
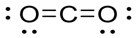
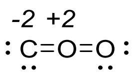
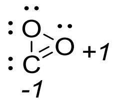
Period / Group	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1	H 1 1s																	He 1 1s
2	Li 1 2s	Be 2											B 1 2p	C 2	N 3	O 4	F 5	Ne 6
3	Na 1 3s	Mg 2											Al 1 3p	Si 2	P 3	S 4	Cl 5	Ar 6
4	K 1 4s	Ca 2	Sc 1 3d	Ti 2	V 3	Cr 4	Mn 5	Fe 6	Co 7	Ni 8	Cu 9	Zn 10	Ga 1 4p	Ge 2	As 3	Se 4	Br 5	Kr 6
5	Rb 1 5s	Sr 2	Y 1 4d	Zr 2	Nb 3	Mo 4	Tc 5	Ru 6	Rh 7	Pd 8	Ag 9	Cd 10	In 1 5p	Sn 2	Sb 3	Te 4	I 5	Xe 6
6	Cs 1 6s	Ba 2	La *1 5d	Hf 2	Ta 3	W 4	Re 5	Os 6	Ir 7	Pt 8	Au 9	Hg 10	Tl 1 6p	Pb 2	Bi 3	Po 4	At 5	Rn 6
7	Fr 1 7s	Ra 2	Ac** 1 6d	Rf 2	Db 3	Sg 4	Bh 5	Hs 6	Mt	Ds	Rg	Cn	Uut	Fl	Uup	Lv	Uus	Uuo

*	Ce 1 4f	Pr 2	Nd 3	Pm 4	Sm 5	Eu 6	Gd 7	Tb 8	Dy 9	Ho 10	Er 11	Tm 12	Yb 13	Lu 14
**	Th 1 5f	Pa 2	U 3	Np 4	Pu 5	Am 6	Cm 7	Bk 8	Cf 9	Es 10	Fm 11	Md 12	No 13	Lr 14

Figure 2. Periodic Table with Valence Electrons Indicated.[2] *Image used under a [Creative Commons Attribution License (by 4.0)] from OpenStax, Chemistry.*

Next, remember the driving force for sharing is achieving a full valence shell for atoms. For main group elements, the full shell contains eight electrons. In Lewis Theory, the octet rule states that an atom will give up, accept, or share electrons to have the filled outer shell. Note hydrogen and helium have a full shell with two electrons since they have a 1s orbital only. To show how the octet rule predicts molecules, Lewis structures illustrate all valence electrons surrounding an atom and how bonding occurs between atoms. Bonds are shown with a line that represents two electrons. Following are a set of steps that will help to get started drawing Lewis structures.

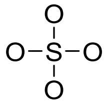
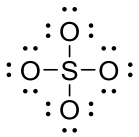
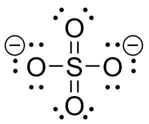
Example: Draw the Lewis structure for carbon dioxide.

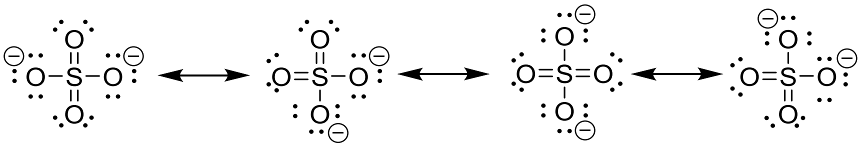
1. Determine the total number of valence electrons in the whole molecule. For formulas with a negative charge, the charge should be added to the number of e- while for formulas with a positive charge, the charge should be subtracted from the number of e-.	Carbon dioxide has the formula CO_2. From the periodic table, C has 4 valence e-, O has 6 valence e-. Sum the e- to obtain the total in the molecule: $1 \times 4e^- + 2 \times 6e^- = 16e^-$
2. Write a plausible Lewis structure by drawing each atomic symbol, and connecting them with line bonds. Often molecules take a symmetrical shape with the less electronegative atom at the central position. Hydrogens will never be central since they only form one bond.	There are a few possible structures for CO_2: O−C−O C−O−O  Let's choose the first since it places the carbon (least electronegative atom) in the center.
3. Count the electrons in the line bonds and determine the remaining electrons to place in the structure. a. Place pairs of electrons as lone pairs around the terminal atoms to give each terminal atom an octet (except hydrogen that only needs two since it has a complete octet with one bond). b. Assign any remaining electrons as lone pairs around the central atom. c. If necessary, move one or more lone pairs of electrons from a terminal atom to form a multiple bond to the central atom. - One shared pair forms a single bond. - Two shared pairs form a double bond. - Three shared pairs form a triple bond.	Each line bond represents two shared e- for 4 e- placed in total: O−C−O Twelve e- remain. Begin to place them on the terminal atoms. Remember that e- are most stable paired:  All 16 e- are placed, so we do not add more. Checking for octet, the oxygens have 8 e- each, but the carbon only has four. Share terminal e- by forming two double bonds: 
4. Finally, to verify the correct Lewis structure, use a formal charge calculation on each atom. *Formal charge =* *(valence e-) – (lone pair e-) –½(bonding e-)* a. Usually, the most plausible Lewis structure is one with no formal charges or very small formal charges. Negative formal charges are most stable on the most electronegative atom. Adjacent atoms should never carry formal charges with the same sign. b. Charges other than 0 should be indicated on the atom since they help to indicate the distribution of electrons on a molecule. c. The sum of formal charges should total the charge on a formula.	Now that all atoms have eight e$^-$, double-check that this is the *best* Lewis structure by using formal charge: *O = (6 val e$^-$) – (4 lp e$^-$) –½(2 bonding e$^-$) = 0* *C = (4 val e$^-$) – (0 lp e$^-$) –½(4 bonding e$^-$)= 0*  Both oxygens have 0 formal charge since they have the same bonding pattern. We can double-check the other possible skeletal structures too:   Since first Lewis structure has 0 formal charges, it is considered the most plausible structure over either of the others above.

There are a few exceptions to the octet rule when drawing Lewis structures. A few atoms tend to have incomplete octets, namely boron, beryllium, and aluminum, since they have few valence electrons. Atoms below period 2 may have expanded octets with 10 or 12 electrons since they have *d* orbitals to use in bonding. In either of these exceptions, following the steps for drawing the Lewis structure and verifying it with formal charge calculations will lead to the most correct Lewis structure.

Occasionally, molecules have more than one reasonable Lewis structure. If these structures have the same connectivity of atoms, but differ in the placement of electrons, they are called resonance structures. The sulfate anion is a good example of both an expanded octet and resonance structures.

Example: Draw the Lewis structure for the sulfate ion.

1. Determine the total number of valence electrons in the whole molecule.	Sulfate ion has the formula SO_4^{2-}. From the periodic table, S has 6 valence e-, O has 6 valence e-, add 2 e- for 2- charge. Sum the e- to obtain the total in the molecule: $1 \times 6e^- + 4 \times 6e^- + 2e^- = 32e^-$
2. Write a plausible Lewis structure by drawing each atomic symbol, and connecting them with line bonds.	Placing the sulfur (least electronegative atom) in the center and radiating the oxygens symmetrically leads to one possible structure for H_2SO_4: 
3. Count the electrons in the line bonds and determine the remaining electrons to place in the structure.	Each line bond represents two shared e- for 8 e- placed in total. Twenty-four e- remain. Begin to place them on the terminal atoms:  All 32 e- are placed, so we do not add more. Checking for octet, all atoms have 8 e- each.
4. Finally, to verify the correct Lewis structure, use a formal charge calculation on each atom.	Now that all atoms have eight e^-, double-check that this is the *best* Lewis structure by using formal charge: $O = (6\ val\ e^-) - (6\ lp\ e^-) - ½(2\ bonding\ e^-) = -1$ $S = (6\ val\ e^-) - (0\ lp\ e^-) - ½(8\ bonding\ e^-) = +2$ While the total of all the formal charges does equal the charge on the ion, the formal charges are too many and too large. We can try to share more e- to see if that helps:  $O = (6\ val\ e^-) - (4\ lp\ e^-) - ½(4\ bonding\ e^-) = 0$ $O = (6\ val\ e^-) - (6\ lp\ e^-) - ½(2\ bonding\ e^-) = -1$ $S = (6\ val\ e^-) - (0\ lp\ e^-) - ½(12\ bonding\ e^-) = 0$ Now, the sulfur has an expanded octet, but the formal charges are smaller throughout the structure. Note the charges indicated on the structure.

	Here, we can see that there are several ways to obtain the same, small formal charges:  Since these structures differ only the position of e-, they are resonance structures.

Molecular models can help with obtaining the correct Lewis structure since the bond positions for each atom are indicated. Additionally, models are important for showing the correct geometry or shape of a molecule.

Geometry can be predicted by Valence-Shell Electron-Pair Repulsion (VSEPR) Theory, based upon the idea that valence electrons in bonded atoms repel one another while being attracted to each nucleus. The theory defines an electron group – a lone pair, a single bond, a double bond, a triple bond, or a single electron (radical) – that creates a negative region in an area of space. The preferred shape is the one that maximizes the separation between these electrons. The mutual repulsions among electron groups lead to electron-group geometry that is the shape of the molecule.

Continuing with the example of carbon dioxide, the double bonds each represent one electron group for a total of two negative regions of space around the central atom. To separate these two electron groups by the maximum distance, they are spaced on opposite sides of the carbon atom – with a bond angle of 180° called a linear geometry (Figure 3).

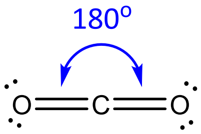
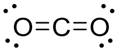
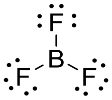
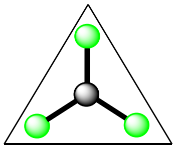
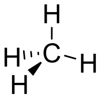
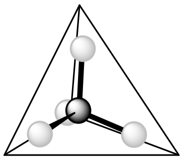
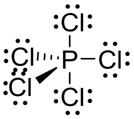
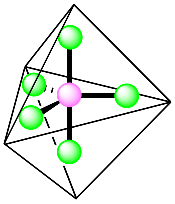
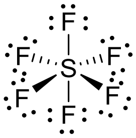
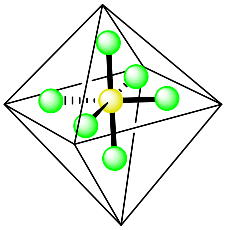


Figure 3. Electron-group Geometry of Carbon Dioxide.

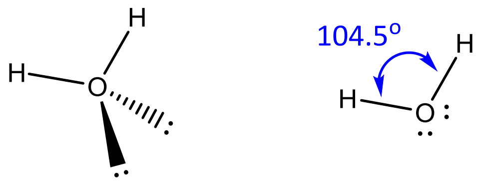
There are five basic shapes that electron groups will adopt thanks to their mutual repulsions: linear, trigonal planar, tetrahedral, trigonal bipyramidal, and octahedral (Table 1). Remembering the polyhedral shapes from which they are named will be helpful in identifying the geometry. Note the use of wedges and dashes to create perspective. A wedge projects toward the viewer while a dash projects back into the page (Figure 4).[3]

Figure 4. Wedge and dash perspective lines.

Table 1. Valence Shell Electron Pair Repulsion Geometries.

Formula	Bonding groups	Lone pairs	Electron-Group Geometry	Bond angles
CO_2	2	0	linear  	180°
BF_3	3	0	trigonal planar  	120°
CH_4	4	0	tetrahedral  	109.5°
PCl_5	5	0	trigonal bipyramidal  	120°/90°
SF_6	6	0	octahedral  	90°/90°

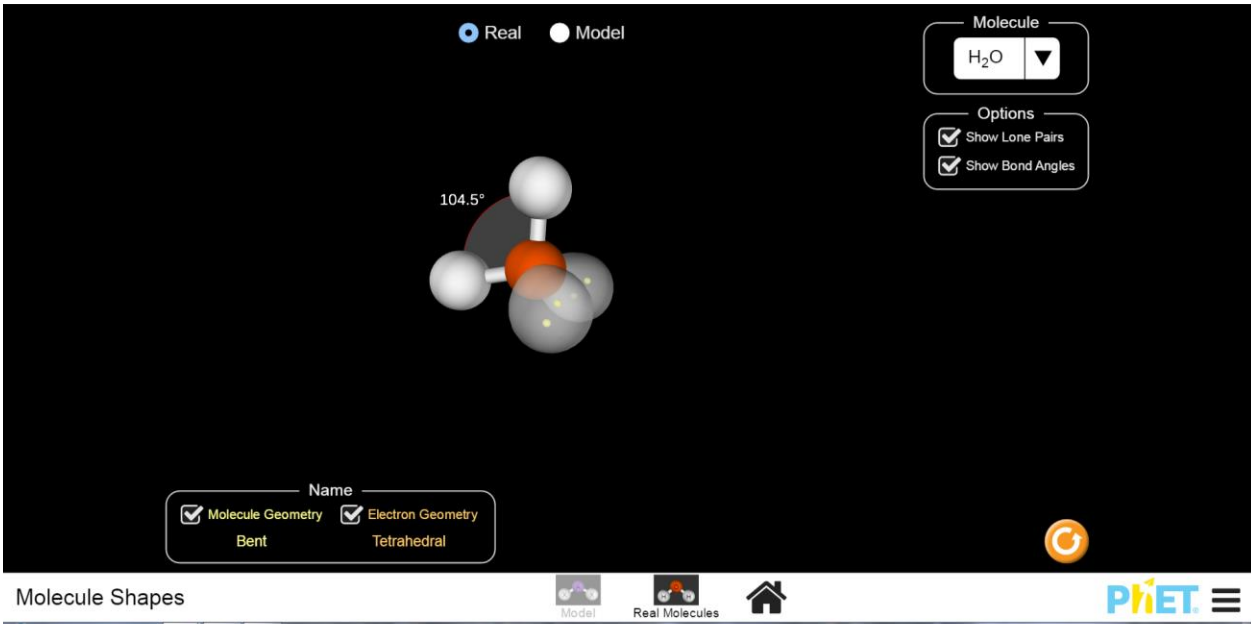
You may be familiar with deviations from the five basic geometries of VSEPR Theory already. For example, water has a bond angle of 104.5° despite having four electron groups, two lone pairs and two single bonds, for which a tetrahedral geometry would be predicted (Figure 5). This decreased bond angle occurs because lone pairs have greater repulsion, being more dispersed, than bonding pairs that are localized between two nuclei.



Tetrahedral electron-geometry, with 109.5° bond angles, is predicted for water with four e- groups, however the actual molecular bond angle is smaller.

Figure 5. Electron-Group vs. Molecular Geometry of Water.

We can use modern simulation software to visualize actual geometries. University of Colorado's PhET Interactive Simulation demonstrates the bond angle differences between the model (theoretical) and real molecular geometry (Figure 6). While the electron-group geometry is still called tetrahedral, the molecular geometry is called bent since visualizing only the atoms leads to a bent line.

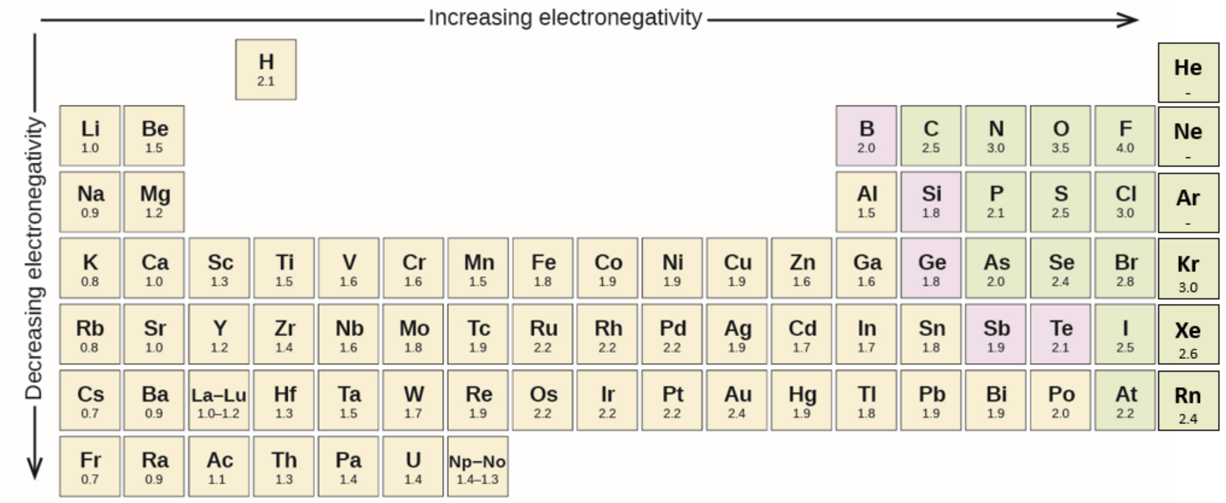


☑ Molecule Geometry: Bent
☑ Electron Geometry: Tetrahedral

Figure 6. University of Colorado PhET Interactive Simulation Demonstrates the Real Shape of Water.[4]

In pre-lab question #1, you are asked to use the PhET Simulation to fill in a table showing how lone pairs affect geometries. *Complete the table now to create a comprehensive illustration of all the molecular geometries.*

Understanding Lewis structures and molecular geometry is important because it allows chemists to predict properties and reactivity of molecules. To complete this understanding, we need to add the concept of bond polarity. While electrons are shared in covalent bonds, they might not be shared evenly. To determine where the electron density lies between two atoms in a bond, we need to review the periodic trend of electronegativity. Atoms have greater electronegativity, the ability to attract bonding electrons to themselves, in the upper right of the periodic table (Figure 7). Atoms at the top of a group are small enough for electrons to experience a stronger attractive force from the protons in the positive nucleus. Atoms on the right side of a period have more protons to create attraction for the electrons than atoms on the left side of a period.



			H 2.1														He -
Li 1.0	Be 1.5											B 2.0	C 2.5	N 3.0	O 3.5	F 4.0	Ne -
Na 0.9	Mg 1.2											Al 1.5	Si 1.8	P 2.1	S 2.5	Cl 3.0	Ar -
K 0.8	Ca 1.0	Sc 1.3	Ti 1.5	V 1.6	Cr 1.6	Mn 1.5	Fe 1.8	Co 1.9	Ni 1.9	Cu 1.9	Zn 1.6	Ga 1.6	Ge 1.8	As 2.0	Se 2.4	Br 2.8	Kr 3.0
Rb 0.8	Sr 1.0	Y 1.2	Zr 1.4	Nb 1.6	Mo 1.8	Tc 1.9	Ru 2.2	Rh 2.2	Pd 2.2	Ag 1.9	Cd 1.7	In 1.7	Sn 1.8	Sb 1.9	Te 2.1	I 2.5	Xe 2.6
Cs 0.7	Ba 0.9	La–Lu 1.0–1.2	Hf 1.3	Ta 1.5	W 1.7	Re 1.9	Os 2.2	Ir 2.2	Pt 2.2	Au 2.4	Hg 1.9	Tl 1.8	Pb 1.9	Bi 1.9	Po 2.0	At 2.2	Rn 2.4
Fr 0.7	Ra 0.9	Ac 1.1	Th 1.3	Pa 1.4	U 1.4	Np–No 1.4–1.3											

Figure 7. Electronegativity Values Calculated by Linus Pauling Demonstrate Increasing Electronegativity to the Upper Right of the Periodic Table.[5]

Once a Lewis structure has been drawn with appropriate molecular geometry, the difference in electronegativity of atoms can be shown to further illustrate the electron density in the molecule. The difference in electronegativity in a bond can be shown two ways: with a cross-base arrow pointing toward the more electronegative atom or with delta plus (δ+) or delta minus (δ-) indicating the charge density (Figure 8).

Figure 8. A Cross-base Arrow or Delta Plus/Minus Symbols Represent Bond Polarity in Hydrogen Chloride.

As electronegativity differences increase between two atoms in a bond, the bond polarity increases until the bond becomes ionic. Nonpolar defines a bond where electrons are shared relatively equally. Polar defines a bond with one electronegative atom that pulls more electron density to itself. Ionic bonds occur most often between a metal and non-metal due to the propensity of the metal to give up electrons to the non-metal, evidenced by the very large electronegativity difference between the atoms in the bond.

Finally, bond polarity combined with geometry defines the polarity of the whole molecule. For example, in carbon dioxide, the individual bonds are polar, however the pull of electrons from each oxygen cancels the other since it is a linear geometry; and the whole molecule is non-polar (Figure 9). In contrast, water has a dipole since an area of charge density can be demonstrated on the oxygen.

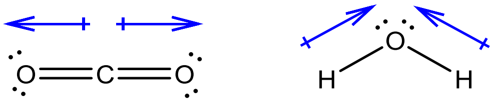


Figure 9. Opposing Polar Bonds in the Nonpolar Carbon Dioxide Molecule and Polar Bonds in Polar Water.

Lastly, we work to define the bond structure, geometry, and polarity in order to predict the physical and chemical properties of the molecule. Dissolution, or dissolving solute into solvent, is an important physical property. The principle of "like dissolves like" is used to predict if a compound will dissolve in a solvent. If two molecules have the same or "like" polarity, they will dissolve together. A polar molecule such as H_2SO_4 will dissolve in a polar molecular such as water. A non-polar compound will dissolve in a non-polar solvent. Polar and non-polar molecules will not dissolve together; for example, in salad dressing, oil and water separate (Figure 10).

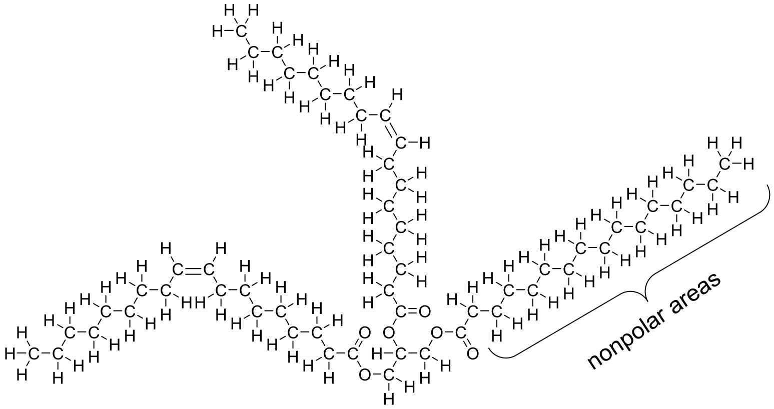


Figure 10. An Example of a Triglyceride, a Molecule Found in Olive Oil, with Large Nonpolar Areas Made of Carbon-to-Carbon and Carbon-to-Hydrogen Bonds That Make It Insoluble with Water.

By combining Lewis Theory, VSEPR Theory, and electronegativity, we can gain a comprehensive illustration of molecular structure. So now, what are your thoughts about the John Ellis Water Machines? Do you think that it is possible to change the natural bond angle of water?

As we have seen, it takes a combination of concepts and several steps to come to an overall picture of a molecule. In this experiment, you will be asked to use ball-and-stick molecular models or the PhET Simulation software to draw Lewis structures, molecular geometries, and then define bond and molecular polarity and solubility.

Experimental Procedure

1. Follow your instructor's directions. Use either the ball-and-stick models or modeling software to help draw accurate Lewis structures and VSEPR geometries, both molecular and electron-geometries. Then, examine the bond polarity and how that affects molecular polarity. Decide if the compound is more likely to dissolve in hexanes, C_6H_{14} a non-polar molecule, or water, H_2O a polar molecule.

2. Draw the tables in your lab notebook, and fill in as required, following the example.

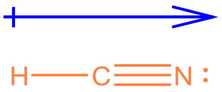
Experiment 11: Molecular Modeling Data

Name: ______________________________ **Date:** __________

Lab Partner: __________________________ **Section:** __________

1)

Formula	**Total Valence Electrons**	**Rough Lewis Structure**	**Molecular Geometry/ Electron-Group Geometry**
HCN	10	H—C≡N : √ octets √ formal charges (include formal charges other than 0.)	H—C≡N : linear/linear

Bond Polarity (ΔEN)	**Molecule Polarity**	**More soluble in hexane or water?**
ΔEN (H – C) = |2.1 – 2.5| = 0.4 ΔEN (C – N) = |2.5 – 3.0| = 0.5		water

2)

Formula	**Total Valence Electrons**	**Rough Lewis Structure**	**Molecular Geometry/ Electron-Group Geometry**
OCl^- *(hypochlorite, oxidizing agent in bleach)*		(include formal charges other than 0.)	

Bond Polarity (ΔEN)	**Molecule Polarity**	**More soluble in hexane or water?**

3)

Formula	Total Valence Electrons	Rough Lewis Structure	Molecular Geometry/ Electron-Group Geometry
NOCl *(nitrosyl chloride, found in 'aqua regia' to dissolve gold)*		(include formal charges other than 0.)	

Bond Polarity (ΔEN)	Molecule Polarity	More soluble in hexane or water?

4)

Formula	Total Valence Electrons	Rough Lewis Structure	Molecular Geometry/ Electron-Group Geometry
$SOCl_2$ *(thionyl chloride, used in chemical synthesis)*		(include formal charges other than 0.)	

Bond Polarity (ΔEN)	Molecule Polarity	More soluble in hexane or water?

5)

Formula	**Total Valence Electrons**	**Rough Lewis Structure**	**Molecular Geometry/ Electron-Group Geometry**
N_2 *(nitrogen, 78% of air)*		(include formal charges other than 0.)	

Bond Polarity (ΔEN)	**Molecule Polarity**	**More soluble in hexane or water?**

6)

Formula	**Total Valence Electrons**	**Rough Lewis Structure**	**Molecular Geometry/ Electron-Group Geometry**
XeO_3		(include formal charges other than 0.)	

Bond Polarity (ΔEN)	**Molecule Polarity**	**More soluble in hexane or water?**

7)

Formula	**Total Valence Electrons**	**Rough Lewis Structure**	**Molecular Geometry/ Electron-Group Geometry**
I_3^-			

Bond Polarity (ΔEN)	**Molecule Polarity**	**More soluble in hexane or water?**

8)

Formula	**Total Valence Electrons**	**Rough Lewis Structure**	**Molecular Geometry/ Electron-Group Geometry**
SiF_4			

Bond Polarity (ΔEN)	**Molecule Polarity**	**More soluble in hexane or water?**

9)

Formula	Total Valence Electrons	Rough Lewis Structure	Molecular Geometry/ Electron-Group Geometry
PH_3			

Bond Polarity (ΔEN)	Molecule Polarity	More soluble in hexane or water?

10)

Formula	Total Valence Electrons	Rough Lewis Structure	Molecular Geometry/ Electron-Group Geometry
BF_4^-			

Bond Polarity (ΔEN)	Molecule Polarity	More soluble in hexane or water?

11)

Formula	**Total Valence Electrons**	**Rough Lewis Structure**	**Molecular Geometry/ Electron-Group Geometry**
$SbCl_6^-$			

Bond Polarity (ΔEN)	**Molecule Polarity**	**More soluble in hexane or water?**

12)

Formula	**Total Valence Electrons**	**Rough Lewis Structure**	**Molecular Geometry/ Electron-Group Geometry**
TeF_4			

Bond Polarity (ΔEN)	**Molecule Polarity**	**More soluble in hexane or water?**

Clean up/Disposal

- If you are using ball-and-stick models, please disassemble them at the end of the lab period, and return to the proper box.
- If you are using a computer, please shut it down, and return to its storage location.

Post-lab

When a molecule has resonance forms, the most stable forms contribute most strongly to the actual structure of the molecule. Stability of resonance structures can be predicted by the following rules:

a) Molecules where all atoms have an octet are more stable.
b) If a molecule has a charge, the more stable structure will have a negative charge on a more electronegative atom, or a positive charge on a less electronegative atom.
c) Molecules with charges separated by smaller distance are more stable than those with larger charge separation.

1. Draw correct Lewis structures for NO_2F. If there is a difference in stability, rank the resonance forms from most (1) to least stable (2).
2. Draw correct Lewis structures for SO_3^{2-}. If there is a difference in stability, rank the resonance forms from most (1) to least stable (3).
3. Draw correct Lewis structures for N_3^-. If there is a difference in stability, rank the resonance forms from most (1) to least stable (3).
4. Include your Experimental Data Tables with your lab report.

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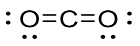
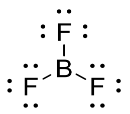
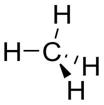
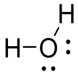
Name: ______________________________________ Date: ____________

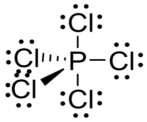
Pre-lab

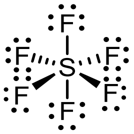
Point your web browser to University of Colorado's PhET Interactive Simulations, and double-click 'Real Molecules' and select water from the drop-down list. Select the radio button 'Show Bond Angles', name 'Molecule Geometry' and 'Electron Geometry'. Using the Molecule Shapes PhET, fill in the table below to create a study tool of molecular geometries of molecules with lone pairs. They can be considered subgroups of electron-group geometries. (Note: you can rotate the molecule by right-mouse clicking on it to get different perspectives of each molecule.)

Include: a) a drawing of the molecule with correct perspective (use wedges and dashes if necessary), b) the name of the molecular geometry and the electron-group geometry, and c) the bond angles around the central atom.

Formula	**Bonding groups**	**Lone pairs**	**Molecular Geometry/ Electron-Group Geometry**	**Bond angles**
CO_2	2	0	linear/linear 	180°
BF_3	3	0	trigonal planar/trigonal planar 	120°
SO_2	2	1		

Formula	**Bonding groups**	**Lone pairs**	**Molecular Geometry/ Electron-Group Geometry**	**Bond angles**
CH_4	4	0	Tetrahedral 	109.5°
NH_3	3	1		
H_2O	2	2	bent/tetrahedral 	104.5°

Formula	Bonding groups	Lone pairs	Molecular Geometry/ Electron-Group Geometry	Bond angles
PCl_5	5	0	trigonal bipyramidal/ trigonal bipyramidal 	120°/90°
SF_4	4	1		
ClF_3	3	2		
XeF_2	2	3		

Formula	**Bonding groups**	**Lone pairs**	**Molecular Geometry/ Electron-Group Geometry**	**Bond angles**
SF_6	6	0	octahedral/octahedral 	90°
BrF_5	5	1		
XeF_4	4	2		

References

1. John Ellis Water®. John Ellis Water Machines. http://johnellis.com/ (accessed March 15, 2017).
2. Image used under a Creative Commons Attribution License (by 4.0) from OpenStax, Chemistry. OpenStax CNX. Mar 10, 2017 http://cnx.org/contents/85abf193-2bd2-4908-8563-90b8a7ac8df6@9.422. Download for free at http://cnx.org/contents/85abf193-2bd2-4908-8563-90b8a7ac8df6@9.422.
3. This tutorial helps to illustrate VSEPR Theory molecular shapes: https://florida.pbslearningmedia.org/asset/lsps07_int_molecularshp/.
4. Image used under a Creative Commons Attribution License (by 4.0). PhET Interactive Simulations, University of Colorado Boulder, February 28, 2017. https://phet.colorado.edu.
5. Image used under a Creative Commons Attribution License (by 4.0) from OpenStax, Chemistry. OpenStax CNX. Mar 10, 2017 http://cnx.org/contents/85abf193-2bd2-4908-8563-90b8a7ac8df6@9.422. Download for free at http://cnx.org/contents/85abf193-2bd2-4908-8563-90b8a7ac8df6@9.422.

VALENCIA COLLEGE

Experiment 12: Laboratory Practical Exam

Version 2

Eileen Pérez, Ph.D., Laura B. Sessions, Ph.D., and Diego J. Díaz López, Ph.D.

This is a formal test, and requires silence and individual work. No questions will be answered regarding experimental procedure or calculations. Safety rules must be followed at all times.

All data must be entered, in pen, directly in the table provided. Do not share calculators.

Materials

List of Chemicals	amount	Equipment	amount
unknown acid	140 mL	50-mL buret	1
standardized NaOH solution	150 mL	125-mL or 250-mL Erlenmeyer flasks	3 minimum
phenolphtalein solution	2 drops per sample	beakers	3 minimum
		small funnel	1
		25-mL pipet	1
		pipet bulb	1
		buret clamp and stand	1
		magnetic bar	1
		magnetic stirring plate	1

Experimental Procedure

1. Add proper labels and headers to the data table on the next page (more columns than needed were provided in case you need to void a trial or want to perform more titrations than requested). Record all data, with the correct number of significant figures in this table as you perform the experiment.
2. Obtain all the equipment and reagents listed above. Record the unknown acid number in the space provided on the next page. The concentration of the NaOH is written on the carboy and on the board.
3. Wash and rinse your equipment using proper laboratory practices.
4. Condition the buret and fill it with the standardized NaOH solution. Read and record the initial volume just before beginning to titrate.
5. Pipet 25.00 mL of the acid solution into an Erlenmeyer flask. Add 2 drops of phenolphthalein. Add a magnetic bar.
6. Place acid sample on magnetic stirring plate and titrate it to the phenolphthalein endpoint. Read and record the final volume of the titrant.
7. Perform at least two more trials. (Remove magnetic bar using the magnet retriever hanging inside of hood.)
8. Transfer all waste to the container in the hood. Rinse the buret and the pipet well and place them in the buret and pipet canisters with the tip up. Wash glassware and return equipment to their proper location.

Calculations

Report the final answer of each calculation with the correct number of significant figures and units of measure.

1. Calculate the concentration of the unknown acid solution in each trial. Report results in the Data Table.
2. Calculate the average concentration of the unknown acid, the standard deviation, and relative standard deviation. Record results in the Results Table with the proper amount of significant figures and unit of measure, if applicable.

The balanced reaction for this titration is: $NaOH(aq) + HA(aq) \rightarrow H_2O(l) + NaA(aq)$

Equations

$$\textbf{standard deviation}, s = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n-1}}$$

$$\% \, RSD = \frac{s}{\bar{x}} \times 100\%$$

CHM 1045C
Laboratory Practical Exam
Data Sheet and Calculations

Name: ______________________________ **Date:** ______________

Data Table: This table has more columns and rows than you need. Properly label as needed, and record all data with the correct amount of significant figures and units.

Calculations & Results: Show calculations of the concentration of at least one trial, calculation of the standard deviation and relative standard deviation of the concentration on the back of this page (you can use the instructions sheet as scratch paper). Include the calculated concentration of the acid in each trial in the table above. Clearly write results in the Results Table below.

Results Table

Unknown Acid Number	
Average Concentration	
Standard Deviation	
% RSD	

For Professor's Use Only: Don't write in this table.		
Grading	**Percent**	**Grade**
Data Table properly labeled, sig. figs.	10 %	
Conc.ea.sample correct & reported	30 %	
Avg. conc. calc. correct, sig. figs. OK	5 %	
Std. dev. calc. correct, sig. figs. OK	10 %	
% RSD calc. correct, sig. figs. OK	5 %	
% Error of Avg. Concentration	30 %	
% RSD <5%	10 %	
Total	**100 %**	