CHEMISTRY 6 AND CHEMISTRY 7 COMBINED LABORATORY MANUAL



Staff
Los Medanos College

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Chemistry 6 and Chemistry 7 Combined Laboratory Manual

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About the Book

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Disclaimer: These experiments are intended to be used with experienced supervision in a laboratory classroom setting. Los Medanos College and the Contra Costa Community College District assumes no responsibility or liability from the use or misuse of these experimental procedures either in or out of the laboratory classroom.

In other words: if you think it is a good idea to go in your back yard (or anywhere else) and mix some random chemicals, we inform you firmly that it is a bad idea. Do not sue us if or when you get hurt. We warned you.

Several sources were used for the experiments in this laboratory manual:

Permission was granted from the Laney College Department of Chemistry to adapt some of their experiments from their 2018 Chemistry 1A, 30A and 30B online lab manuals. Experiments 2, 5, 6, 7, 8, 9, 10, 12, 13, 15, 17, 18, 19, and 20 were adapted for use from Chemistry 1A. Experiment 3 from Chemistry 30A and Experiments 3, 9, and 11 from Chemistry 30B were also adapted.

Permission was granted from the Santa Monica College physical science department to adapt their experiments from their Chemistry 9 and Chemistry 10 laboratory manuals. The Chemistry 9 experiments “Double Displacement Reactions”; “Flame Tests and Atomic Spectra”, “Acids, Bases and pH”, “Making Soap”; “Lewis Structures and Molecular Shapes”; and “Electrical Conductivity of Aqueous Solutions” were adapted for use. The Chemistry 10 experiment “Experiment 12: Equilibrium and LeChatelier’s Principle” was also adapted for use.

The 2018-2020 staff of student stockroom workers were of great assistance in testing out and improving these experiments. Listed in alphabetical order they are Most Akter, Jacqueline Abad Santos, Elijah Avery, Shanice Blake, Estrella Casteneda, Diane Clark, Ashley Delatorre, Anthony Early, Abbey Ishola, Andreana Gomez, Kole Holland, Stephen Kohl, Brenda Kwan, Neha Narayan, Nicolette Moss, Gabriana La, Emmie Reed, Tennile Richardson, Stone Silva, Glen Stafford, Abdiel Torres, and Jacqueline Villalobos.

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Effort was made to acknowledge all contributors to this document. If anyone was inadvertently omitted, please contact LMC and a correction will be made.

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ExperimentList

Experiment	
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Technique	
Letter	**Title**
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C	Use of Balances
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F	Crucible use
G	Buret use
H	Use of the Bunsen burner
I	Use of Spec 20/Spec 200

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Organization_Version_Control_1_2

Laboratory Manual Organization

This laboratory manual is organized in three parts:

Part 1 is laboratory experiments. These are available to instructors for laboratory class assignments. There are more experiments than are necessary for most classes, so students may not do all experiments in this manual.

Part 2 is laboratory techniques. These techniques are sometimes used in multiple experiments. They are available for both reference and supporting instructions on laboratory experiments.

Part 3 is for supplemental documents. This section is for more detailed explanations, additional problems sets, and abridged reporting sheets for specific experiments. This section is intended for faculty to add customized supplemental documents.

Experiments are numbered in the sequential order they were prepared. There is no particular relevance to the sequential numbering scheme. Experiments primarily intended for the Los Medanos College Chemistry 06 course begin with a '6'. Experiments primarily intended for the Chemistry 07 course begin with a '7'. Faculty may assign experiments as appropriate across both courses. Refer to the appropriate course syllabus for which experiments are being assigned by your instructor.

Pagination is done within each experiment and technique.

Version Control

Because this laboratory manual is intended to be improved over time, all documents use version control. Users of this manual should be aware of how version control has been implemented. In the upper right hand area of all pages, there is a version number. Version numbers are changed when a revision is done, and are also incorporated into the filenames of the individual experiments. The magnitude of the difference done during an update will determine how much the version number is changed.

A minor change such as a misspelled word would cause the version to change in the third place. For example, if an experiment went from version 1.3.2 to 1.3.3, it indicates a minor change between the two versions.

A moderate change, such as adding a section or changing a diagram or photograph would cause the version to change in the second place, and resetting the third place. For example, a technique changed from version 1.2.5 to version 1.3 indicates that a significant change occurred, but that the underlying procedure is similar.

A major change, such as a full rewrite of the procedure, would be a large and significant change. For example, a change from version 2.3.4 to version 3 would indicate that the two versions are fundamentally different.

The most current version of experiments and techniques will be offered as the individual file (available for uploading to Canvas). An annual update is planned for a single production file for easy printing.

REVISION HISTORY

Section	Version Number	Major Change(s)
Summer 2020		
Title Page	June 2020	Updated to new revision.

Version Control	1.2	Revision History Table added.
Experiment List	June 2020	Added Exp 731: Esters. Misspelled word fixed
Experiment 601	1.6	Revised
Experiment 607	1.1.2	Minor revision. Changed 'Replacement' to 'Displacement'
Experiment 611	2.0	Revised
Experiment 615	1.2.2	Minor revisions
Experiment 625	1.1.3	Revised
Experiment 729	1.2	Revised
Experiment 731	1.2.1	New

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CHAPTER 1: EXPERIMENTS

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1.1: Experiment_601_Measurement 1_6

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 601: Measurement & Dimensional Analysis**

Experiment 601: Measurement & Dimensional Analysis

Section 1: Purpose and Summary

- Describe how the English and metric systems of measurement compare.
- Learn how to convert between different units of measurement.

In this experiment, students will practice measuring the following parameters: length, mass, volume, and temperature. Students will calculate derived constants including density and heat capacity. Students will practice converting from units in the English system to the metric system. Students will learn dimensional analysis, common prefixes and selected conversion factors.

Note: When a parameter is determined, it needs to have both a number and the unit of measurement to be complete. If you only write down the number “5”, for example, is it “5 inches”, “5 centimeters”, “5 meters” or “5 miles”?

Note: When writing decimals between zero and 1, a good practice is to include the leading ‘0’ before the decimal point. For example, if you write “**.**125” centimeters onto your data report, it is possible that you might accidentally misinterpret the value as 125 centimeters. But if you write “**0.**125” centimeters there should be no question that there is a decimal point present.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

All of the materials in this experiment are harmless. Use of eye protection is recommended for all experimental procedures.

Waste Disposal:

No waste will be generated for this experiment.

Section 3: Procedure

Part 1: LENGTH

1. Using a large ruler, carefully measure the width of your lab bench in **centimeters** and in **inches**. Remember to use proper significant figures.

2. These two measurements allow us to create a pair of conversion factors between centimeters and inches. A conversion factor (also called a unit factor) is a ratio which can be multiplied by a measurement expressed in one set of units, to convert the measurement to a new set of units.

To find a conversion factor for converting **centimeters to inches**, divide **measurement (b)** by **measurement (a)**:

$$\frac{(b)\quad in}{(a)\quad cm} = \text{in/cm}$$

3. To find a conversion factor for converting **inches to centimeters**, divide **measurement (a)** by **measurement (b):**

$$\frac{(a)\quad cm}{(b)\quad in} = \text{cm/in}$$

4. Obtain a numbered card with a line of unknown length.

Measure the line to the tenth of a centimeter (0.1 cm), and convert this measurement to inches **using the appropriate conversion factor** from your work in Step 2, above.

Show your work here:

$$\frac{(e)\quad cm}{1} \times \frac{\qquad\qquad}{\qquad\qquad} =$$

Hint: Any quantity can be written as a fraction over 1. If the units cancel, you have selected the proper conversion factor.

5. Measure the height of your lab partner in inches. Convert this measurement to centimeters using the appropriate conversion factor from Step 3. Remember to include the units on each quantity.

Show your work here:

Part 2: MASS

1. Obtain one of the numbered metal slugs. Measure and record its mass to the **hundredth of a gram (0.01 g).** Keep this metal slug for volume measurement in Part 3 of this experiment.

2. Using the appropriate conversion factors from **Tables I and II** (page 8)**,** convert the mass of the slug into **ounces, pounds, kilograms and milligrams:**

Show your work here:

$$\frac{(a)}{1}\ \frac{grams}{} \times \frac{ounces}{grams} = (b)\ \text{ounces}$$

$$\frac{(b)}{1}\ \frac{ounces}{} \times \frac{pounds}{ounces} = (c)\ \text{pounds}$$

$$\frac{(a)}{1}\ \frac{grams}{} \times \frac{kilograms}{grams} = (d)\ \text{kilograms}$$

$$\frac{(a)}{1}\ \frac{grams}{} \times \frac{milligrams}{grams} = (e)\ \text{milligrams}$$

Part 3: VOLUME

1. Fill a measuring cup with tap water, so that the bottom of the meniscus rests exactly on the 12-ounce mark. Give your best estimate of how many **milliliters (mL)** this represents.

NOTE: Because the symbol for liters is both “l”, and “L”, the symbol for milliliters can be either “ml” or “mL”. When taking notes, use whichever symbol provides the clearer meaning and be consistent.

2. Use your measurements to calculate a conversion factor from **ounces to milliliters** and **milliliters to ounces.**

Show your work here:

$$\frac{12 \quad oz}{(a) \quad mL} = \text{(b)} \qquad \text{oz/mL}$$

$$\frac{(a) \quad mL}{12 \quad oz} = \text{(c)} \qquad \text{mL/oz}$$

3. A large bottle of beer contains 22 ounces of fluid. Using the appropriate conversion factor from Step 2 above, convert the volume of this beer into **milliliters.**

Show your work here:

$$\frac{22 \quad ounces}{1} \times \frac{ml}{ounce} = \text{(d)} \qquad \text{milliliters}$$

Using the appropriate conversion factor from **Table II** (page 8), convert the volume of this beer into **cups, gallons, and microliters.**

Show your work here:

$$\frac{22 \ ounces}{1} \times \frac{cups}{ounces} = \text{(e)} \qquad \text{cups}$$

$$\frac{(e) \quad cups}{1} \times \frac{quarts}{cups} \times \frac{gallons}{quarts} = \text{(f)} \qquad \text{gallons}$$

$$\frac{(d) \quad mL}{1} \times \frac{L}{mL} \times \frac{microliters}{L} = \text{(g)} \qquad \text{microliters}$$

NOTE: The symbol for micro is the Greek letter mu, “μ”

4. Take your metal slug from Part 2 of this experiment. Fill a 50-mL graduated cylinder with tap water to about the 35-mL mark. Record the exact volume of water.

5. Tilt the graduated cylinder and carefully slide the metal slug into the cylinder. Record the final volume of water.

6. The volume of the metal slug is equivalent to the amount by which the water level rises when the metal is submerged. Calculate the volume of the metal slug: (i) – (h)

Part 4: DENSITY

The mass of a substance divided by its volume is called its **density,** thus

Density= $\frac{\text{mass}}{volume}$

1. Using the mass and the volume of the metal slug you measured in Parts 2 and 3, calculate its density in units of **g/mL**?

Show your work here:

2. The density of the metal slug that you calculated in Step 1 above can be used as a conversion factor.

What would be the mass in grams of 10. mL of this substance?

$$\frac{10.}{1}\ \frac{mL}{} \times \frac{grams}{mL} = \text{(b)}\ \text{grams}$$

3. What volume, in milliliters (mL), would be occupied by 375 grams of this metal?

Show your work here:

NOTE: Density is *not* a quantity, but a property of a material. A mass of an item depends on the quantity of matter in it. The density is how compact that matter is within the space it occupies. The units on both are different.

Part 5: TEMPERATURE & SPECIFIC HEAT

1. Remove your Celsius (centigrade) thermometer from its protective case and let it sit for two minutes at room temperature. Record the temperature of the room. Add 300 milliliters of tap water to your 600 mL beaker. Using the apparatus demonstrated by your instructor, bring this water to a rolling boil. Measure the temperature of the boiling water and record this value.	Room temperature: (a) _____°C Boiling water temperature: (b) _____°C
2. Temperatures in the units of Celsius can be converted to units of Fahrenheit by the formula: $(\frac{9}{5} \times ℃) + 32 = ℉$ Likewise, Fahrenheit temperatures can be converted to Celsius as follows: $\frac{5}{9} \times (℉ - 32) = ℃$ Using the appropriate formula, find the temperature of the room in degrees Fahrenheit. Show your work here:	Room temperature: (c) _____ °F

3. The amount of **energy** needed to raise the temperature of **one gram of substance by one degree** is called its **specific heat.** For example, the specific heat of water can be expressed as

1.00 calorie /g°C (or 4.18 Joules/g°C) whereas the specific heat of glass is 0.20 calorie/g°C (or 0.84 Joules/g°C).

Whenever a substance is heated from a lower to higher temperature, or *vice versa*, the amount of energy absorbed from the surroundings (to heat it) or released to the surroundings (as it cools) can be calculated by this general formula:

energy exchanged = (mass of substance)(specific heat)(temperature change)

The temperature change, usually called delta-T or "ΔT" is simply the final temperature minus the initial temperature:

FIX

$\Delta T = T_{final} - T_{initial}$

where T final is the final temperature, and T initial is the initial temperature. Note that ΔT can be either positive or negative.

When water is being considered, and we use units of calories, the equation becomes:

calories exchanged = mass × $\frac{1.00cal}{g°C}$ ×(ΔT)

Let us calculate the number of heat calories absorbed by the water from the Bunsen burner when you boiled it in Step 1 above. We will assume that the water started out at room temperature (even though that might not really be the case).

(d) ΔT = °C - °C = °C

(e) If the density of water is 1.00 g/mL, what is the mass of 300. mL of water?

Show your work here:

(f) Using your calculated values from parts (d) and (e), determine the number of calories absorbed by your heated water:

grams of water *x* $\frac{1.00cal}{g°C}$ ×(ΔT) *x* ____°C = cal

Note: There are 3 units required for specific heat: one unit in the numerator (calorie or Joules) and 2 units in the denominator (grams and Celsius).

(d) ΔT = ^{o}C

(e) grams

(f) calories

TABLE I: COMMON PREFIXES USED IN THE METRIC SYSTEM

	Giga, G	=	1,000,000,000	=	10^9				

	Mega, M	=	1,000,000	=	10^6				
	kilo, k	=	1,000	=	10^3				
	deci, d	=	0.1	=	10^{-1}	=	1/10		
	centi, c	=	0.01	=	10^{-2}	=	1/100		
	milli, m	=	0.001	=	10^{-3}	=	1/1,000		
	micro, μ	=	0.000001	=	10^{-6}	=	1/1,000,000		
	nano, n	=	0.000000001	=	10^{-9}	=	1/1,000,000,000		

TABLE II: SELECTED CONVERSION FACTORS

Length									
	12 inches (in) = 1 foot (ft)		3 feet = 1 yard (yd)				5,280 feet = 1 mile (mi)		
Mass									
	16 ounces (oz) = 1 pound (lb)			28 grams (g) = 1 ounce (oz)					
Volume									
	8 ounces* = 1 cup		16 ounces* = 1 pint				4 cups = 1 quart		
	4 quarts = 1 gallon								

* The English/American system of measurements uses ounces for both mass and volume. When ounces are used for volume measurements, they are usually written as “fluid ounces”, or “fl. oz.” There is no easy comparison or conversion between ounces used for mass and fluid ounces used for volumes.

Post Lab Questions:

1. An older unit of length used in the English system is the *rod*. A rod is equal to 16.5 feet. Calculate the number of centimeters that are in 1 rod. Some of the setup for your calculation is below:

$$\frac{1 \quad rod}{1} \times \frac{feet}{rod} \times \frac{inches}{foot} \times \frac{2.54}{1}$$

=_____

2. In medicine, the *grain* was used as an older unit of measurement. One grain is equal to 64.79891 milligrams (mg). Often, a 5-grain dosage of aspirin would be prescribed.

Calculate the milligram dosage of a 5-grain tablet of aspirin.

$$\frac{5 \quad grain}{1} \times \frac{mg}{grain} = \text{milligrams of aspirin.}$$

What is a common dosage of aspirin in a tablet for adults? (Search the internet for an answer.) How does it compare with the traditional amount delivered as 5 grains?

3. The *karat* is a fractional measure of purity for gold. Pure gold is defined to be 24 karats. So 24 karat gold would be (24/24), or 100% gold. 18 karat gold would be 18/24 or 75% gold, with the remainder a different metal in the alloy. The different metal is generally much cheaper than the gold and the cost of that metal can be ignored.

You are on travel in a foreign country. The local unit of currency is the *solar*. The current exchange rate is 3.26 *solars* are equivalent to $1.00 (U. S. dollar).

You see a very attractive piece of jewelry that is 14 karat gold. It weighs 25 grams. The spot price of gold today is $38.06 per gram.

The vendor is asking 1200.00 *solars* for the jewelry. Is this a good deal (based on the value of the gold content)? Show your work.

Notes:

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1.2: Experiment_602_Empirical Formula of MgO 1_4_2

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 602: Empirical Formula**

Experiment 602: Empirical Formula

Section 1: Purpose and Summary

- Determine the empirical formula of magnesium oxide.
- Calculate the mass of oxygen using the weighing-by-difference method.
- Calculate the moles of a sample from its mass.

In this experiment, students will conduct the reaction between magnesium and oxygen gas. Students will determine the mass of the magnesium sample before and after the reaction, as well as the mass of magnesium and oxygen in the product. Students will learn how to convert mass to moles of a given sample and determine the empirical formula of a substance from mass and mole data.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Do not look directly at the burning magnesium ribbon. The flame is bright enough to damage your eyes. Use of eye protection is required for all experimental procedures.

A hot crucible will break if placed directly on a cold surface. Set hot crucibles onto wire screens to cool.

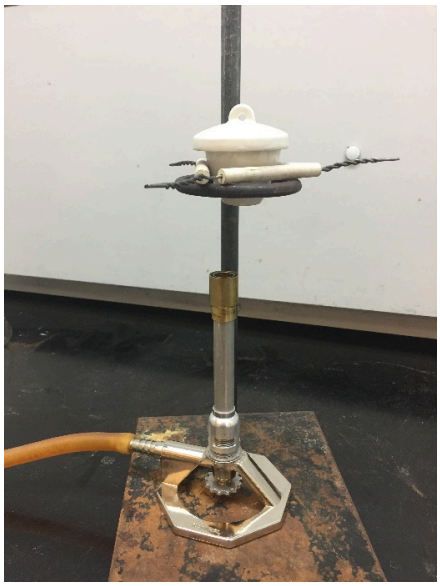
A hot crucible will break if splashed with water directly. Let the crucibles cool before adding water.

Waste Disposal:

The solid product from the reaction can be disposed of in the regular waste bin in the lab.

Section 3: Procedure

Part 1: Preparation of the crucible

1. Heat a clean and dry porcelain crucible with lid (on a clay triangle supported on a ring stand) directly over a Bunsen burner flame for about 5 minutes. This will evaporate water and burn off any greasy organic impurities, ensuring the crucible is clean and dry. Here is what the setup should look like: 	
2. Turn off the burner and allow the crucible to cool. Leave the crucible resting on the clay triangle. **DO NOT** put a hot crucible on the lab bench.	
3. When the crucible is cool to touch, weigh the crucible and lid on the digital balance. **It is important to use a balance that measures to 0.0001 grams. If you use the wrong balance, you will have to repeat this experiment.**	Mass of empty crucible and lid: (a)_____ grams

Part 2: Preparation of magnesium sample

1. Obtain a strip or two of magnesium ribbon, approximately 6.0 to 7.0 cm in length. NOTE: Magnesium has a shiny silver-white surface, but may lose its shine by reacting with oxygen in the air, forming a magnesium oxide coating. If it is not shiny, polish it slightly with steel wool to remove any oxide coating. Use sufficient magnesium strips to mass between 0.4 and 0.6 grams on an analytical balance (a balance that measures to 0.0001 g)	
2. Fold or loosely coil the magnesium ribbon(s) to make sure they sit entirely on the bottom of the crucible.	

3. Place the coiled magnesium ribbon into the crucible, cover it, and weigh it on the digital balance. Record mass.	Mass of crucible, magnesium ribbon, and lid: (b)_____ grams

Part 3: Heating the magnesium sample

1. Place the crucible containing the magnesium ribbon back into the clay triangle. Put the lid close by on the lab bench, with crucible tongs to handle it later.	
2. Light the Bunsen burner and start heating the crucible and magnesium ribbon. As soon as the magnesium ribbon starts to burn, replace the lid (using crucible tongs) to extinguish the flame. An unbalanced equation of magnesium burning in air is the following: $\mathrm{Mg\,(s) + O_2\,(g) \longrightarrow Mg_xO_y\,(s) + Mg_3N_2\,(s)}$, where $\mathrm{Mg_3N_2}$ is a by-produced, and x and y in the oxide of magnesium are positive integers. Some of the oxide particles may be lost as smoke if the lid is not placed over the crucible.	
3. Continue heating the crucible with the lid on for a minute or so, then remove the lid again to replenish the oxygen consumed. When the magnesium ribbon starts to burn again, replace the lid to extinguish the flame.	
4. Repeat this heating process until the magnesium ribbon no longer catches fire.	
5. With the lid on, heat the crucible and sample strongly for 5 minutes. Make sure that the bottom of the crucible turns 'red' hot.	
6. Turn off the burner, and let the crucible, lid, and sample cool on the clay triangle.	
7. When the crucible is cool to touch, remove the lid, then add about 10 drops of laboratory water into the burned magnesium ribbon. Ensure that the entire sample is wet, not just one spot. Water reacts with the by product $\mathrm{(Mg_3N_2\,(s) + 6\,H_2O\,(l) \longrightarrow 3\,Mg(OH)_3\,(s) + 2\,NH_3\,(g)}$	
8. Light the Bunsen burner and heat the crucible (without a lid) with a low flame for about a minute. This evaporates the surplus water you just added. After the water has evaporated, increase the flame and heat it strongly for about 10 minutes. The bottom of the crucible does not need to turn 'red' hot during this heating. This heating converts $\mathrm{Mg(OH)_3}$ into magnesium oxide by the following unbalanced equation: $\mathrm{Mg(OH)_3\,(s) \xrightarrow{heat} Mg_xO_y\,(s) + H_2O\,(g)}$	
9. Turn off the burner, and let the crucible and the sample cool on the clay triangle.	

10. When the crucible is cool to touch, weigh the crucible, product, and lid on the digital balance.	Mass of crucible, product, and lid: (c)_____ grams

Section 4: Calculations

1. From the masses you recorded in Parts 1 and 2 of this experiment, calculate the mass of the magnesium ribbon.

(b) – (a)

2. From the masses you recorded in Parts 2 and 3 of this experiment, calculate the mass of magnesium oxide produced from the reaction.

(c) – (a)

3. From the mass of magnesium oxide (e) and the mass of magnesium ribbon (d), calculate the mass of oxygen that combined with magnesium during the reaction.

(e) – (d)

4. Refer to a periodic table to obtain the molar mass of magnesium. Then, using this information and the mass of magnesium ribbon (d), calculate the number of moles of magnesium that you started with.

Molar mass of Mg: _____g/mol (from a Periodic Table)

Show your equation here:

EXAMPLE: If a student used 0.4532 g of Mg, then set up your equation like this. Use your molar mass of Mg value. Set up the equation so that the units cancel.

$$\frac{0.4532\ grams\ \ Mg}{1} \times \frac{moles\ of\ Mg}{grams\ of\ Mg} = \qquad mol\ of\ Mg$$

5. Obtain the molar mass of oxygen from a periodic table. Using this information and the mass of oxygen you calculated **(f)**, calculate the number of moles of oxygen that combined with the magnesium.

Molar mass of O: _____ g/mol (from a periodic table)

Show your equation here:

EXAMPLE: If a student measured a mass gain of 0.3005 g due to the combustion of oxygen, then set up your equation like this. Use your molar mass of oxygen value. Set up the equation so that the units cancel.

$$\frac{0.3005\ \text{grams}\ \ \text{O}}{1} \times \frac{\text{moles of O}}{\text{grams of O}} = \text{mol of O}$$

6. Calculate the mole ratio between magnesium and oxygen by dividing the mol Mg **(g)** by the mol O **(h)**.

Show your equation here:

EXAMPLE: If a student measured *a* moles of Mg and *b* moles of O, the equation should be set up as Set up the equation so that the units cancel.

$\frac{\text{a}}{\text{b}} \frac{\text{moles of Mg}}{\text{moles of O}}$ = mole ratio of Mg to O in magnesium oxide

7. Using your mole ratio determined in (i), write the empirical formula of magnesium oxide by replacing x and y in $\mathrm{Mg}_x\mathrm{O}_y$ with positive integers. Round off the ratio to the nearest whole number to obtain the x and y integers.

Post Lab Questions:

1. Some experimental errors could result in a high or low molar ratio between Mg and O. In each case below, decide whether the situation described would lead to a calculated ratio of too much oxygen or too little oxygen, and explain your answer.

(a) You forgot to perform the initial drying step and proceeded directly to weighing the crucible and lid you obtained from the stockroom.

(b) Your magnesium ribbon is not shiny. But you did not polish it with steel wool before use, as indicated in the experiment.

(c) You added more laboratory water than is needed in Part 3 Step 7, and you did not dry it out completely.

(d) After strong heating of the crucible, you removed the lid but dropped it and broke it. You then obtained a new lid for the final weighing.

2. A similar experiment is performed to determine the empirical formula of an oxide of copper, and the following data were collected. Predict the empirical formula of copper oxide from these data.

Mass of crucible, cover, and copper sample: 21.53 g

Mass of empty crucible with cover: 19.66 g

Mass of crucible, cover, and sample (after heating): 21.76 g

How can the experiment for the determination of the empirical formula of an oxide of copper be improved?

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1.3: Experiment_603_Separating Components of a Mixture 1_4_3

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 603: Separating Components of a Mixture**

Experiment 603: Separating Components of a Mixture

Section 1: Purpose and Summary

- Learn how to separate components of a mixture.
- Calculate the percent composition of a mixture.
- Calculate percent recovery of sample.

In this experiment, students will separate the components of a mixture containing sand (mostly SiO_2), table salt (NaCl), and calcium carbonate ($CaCO^3$). Students will perform various separation techniques such as dissolution, filtration, decantation, and evaporation to isolate each component of the mixture. Students will learn how to calculate the mass of recovered component after each separation and the determine percent recovery of a sample.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Hydrochloric acid (HCl) is corrosive and toxic. Avoid contact with your eyes, skin, and clothing.

Handle hot glassware with tongs.

Do not place hot glassware on cold or wet surfaces. The thermal shock can break glass and porcelain.

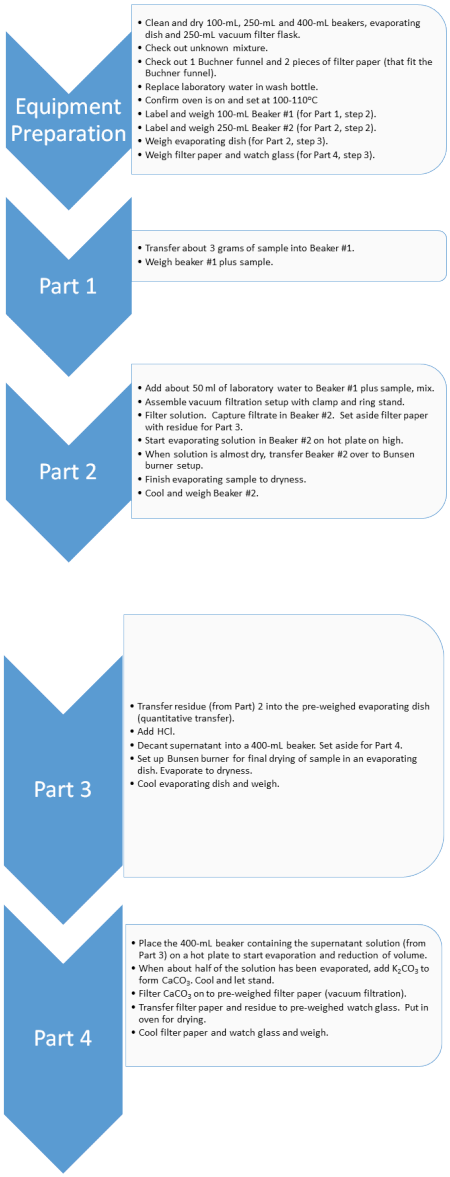
Use of eye protection is required for all experimental procedures.

Waste Disposal:

Collect all recovered materials in a designated waste beaker.

Section 3: Procedure

NOTE: Time management is extremely important to be able to successfully complete this experiment within one laboratory period. Use the following flow chart:



Part 1: Preparation of the sample mixture

1. Obtain an unknown sample from the stockroom window. **Record the unknown sample number.**	Unknown sample number:
2. Weigh a **clean and dry** 100-mL beaker (Beaker #1).	Mass of Beaker #1: (a) _____grams
3. Transfer approximately 3 grams of unknown sample into Beaker #1. Record the exact mass to the nearest centigram (two decimal places).	Mass of Beaker #1 with unknown sample: (b) _____grams

Part 2: Extraction of NaCl

1. To the sample, add approximately 50 mL of laboratory water and stir continuously with a glass stirring rod for about two minutes. Let mixture stand for a few minutes to allow the undissolved components settle to the bottom of the mixture.	
2. Weigh a **clean and dry** 250-mL beaker (Beaker #2). Record exact mass.	Mass of Beaker #2: (c) _____grams
3. Weigh a **clean and dry** ceramic evaporating dish. Record exact mass.	Mass of evaporating dish: (d) _____grams
 4. Prepare a vacuum filtration set up: a) You will need a Buchner funnel, a clean 250-mL vacuum filter flask, a filter adaptor (often a one-hole stopper), a pre-cut filter paper and Beaker #2. b) Place the filter paper on the Buchner funnel. The filter paper should fit snugly and cover all the small holes in the funnel. c) Using the filter adaptor, place the Buchner funnel on top of a vacuum filter flask. You might need to use a clamp and a ring-stand to keep the setup intact and upright. d) Connect a vacuum tubing hose to the vacuum filter flask and to the vacuum supply. e) Wet the filter paper with a little laboratory water. Turn the vacuum on and confirm that there are no leaks.	

5. While the vacuum is engaged, carefully pour the water/sample mixture into the funnel. Transfer all the residue into the funnel. Use some laboratory water if necessary, to transfer the residue.	
6. Gather the filter paper from the funnel using the edge of the filter paper. You might need to use tweezers. Avoid losing any solids. Save the solids and the filter paper in the pre-weighed evaporating dish for Part 3.	
7. You need to transfer all the filtrate from the vacuum filter flask to another container to evaporate it to dryness. This is called a *quantitative transfer*. Pour the filtrate from the vacuum filter flask into Beaker #2. Using a minimal amount of laboratory water, rinse the inside of the vacuum filter flask into Beaker #2. Three good rinses is usually sufficient.	
 8. Begin the evaporation of the solution in Beaker #2 on a hot plate. Do not evaporate to dryness on the hot plate. You will have better control of the final evaporation by using a Bunsen burner setup.	
9. When sample in Beaker #2 is almost dry, place Beaker #2 on a wire mesh and ring stand over a Bunsen burner. Heat the solution in Beaker #2 on a Bunsen burner and evaporate to dryness. As the solution approaches dryness, it may sputter if it is heated too fast. Avoid loss of product by finishing this final evaporation step slowly.	
10. After the sample has evaporated to dryness, allow Beaker #2 to cool and weigh. Record exact mass.	Mass of Beaker #2 with residue (NaCl): (e) _____grams
11. Wash, rinse, and dry Beakers #1 and #2.	

Part 3: Separation and recovery of sand

1. Using a spatula, completely transfer the residue from the filter paper into the pre-weighed evaporating dish. Wash the filter paper with a few drops of laboratory water to complete the transfer. When you are satisfied that no solids remain on the filter paper, discard the filter paper.	
2. To this mixture of solid sample and water, **slowly** add about 8 mL of 3 molar (M) hydrochloric acid (HCl) solution. Stir the mixture continuously until the bubbling disappears. Let the mixture stand for a few minutes to settle.	
3. Decant the supernatant into a 400-mL beaker, collecting as much liquid as you can. Avoid transferring any remaining solids from the evaporating dish. Wash the residue in the evaporating dish with approximately 5 mL of laboratory water; allow the residue to settle and decant again. Do the washing and decanting steps two times, collecting all liquid into the 400-mL beaker. Avoid transferring any solid material to the beaker.	
4. After the last washing is completed, set aside the liquid in the 400-mL beaker for Part 4 of this experiment.	
5. Set up a Bunsen burner with a wire screen on a ring stand with an iron ring. Place the evaporating dish on the wire screen and use the Bunsen burner to drive off all moisture from the residue. Evaporate any liquid until the residue on the evaporating dish is dry.	
6. Cool the evaporating dish and weigh. Record exact mass.	Mass of evaporating dish with residue (sand): (f) _____grams
7. Discard sand into the garbage can, NOT in the sink.	

Part 4: Extraction of $CaCO_3$

1. Using a hot plate, evaporate the solution in the 400-mL beaker to boiling. Continue boiling for 5 minutes or until about half of the volume of the solution remains.	
2. Using tongs, remove the 400-mL beaker from the hot plate. Let it rest on a wire gauze on the bench top and immediately add about 15 mL of 1 M K_2CO_3 solution. Stir the mixture for about 5 minutes, then allow it to cool to room temperature. This delay allows small microcrystals to become larger, which makes it possible to filter them.	
3. Meanwhile, obtain another pre-cut filter paper and a clean, dry watch glass. Put the filter paper on the watch glass and weigh. Record exact mass.	Mass of watch glass and filter paper: (g) _____grams
4. Prepare a vacuum filtration set up, as described in Part 2 of this experiment. Use the pre-weighed filter paper above (step 3).	
5. Carefully pour the reaction mixture into the Buchner funnel. Transfer all the residue ($CaCO_3$) into the funnel using a stream of laboratory water from a wash bottle.	
6. After filtration is done, discard filtrate into the drain. Remove the filter paper containing $CaCO_3$ and place it on the pre-weighed watch glass.	
7. Place the filter paper and $CaCO_3$ on the watch glass in an oven to dry completely. If the oven temperature is at 100 °C, this should take around 15 minutes. Be careful to use tongs to remove the watch glass from the oven. It will be hot! Alternatively, your instructor may allow you to let your $CaCO_3$ air dry and weigh it during the next laboratory period.	
8. If you used the oven method for drying, let the watch glass cool to room temperature. Weigh the dried $CaCO_3$, filter paper and watch glass. Record exact mass.	Mass of watch glass, filter paper, and $CaCO_3$: (h) _____grams
9. Discard filter paper and residue into the garbage can in the lab.	

Section 4: Calculations

1. From the masses you recorded in Part 1 of this experiment, calculate the **mass of your unknown sample mixture**. **(b) – (a)**	Total sample mixture (by difference) (i) _____ g
2. From the masses you recorded in Part 2 of this experiment, calculate the **mass of NaCl extracted**. **(e) – (c)**	(j) _____ g NaCl
3. From the masses you recorded in Part 3 of this experiment, calculate the **mass of sand recovered**. **(f) – (d)**	(k) _____ g sand
4. From the masses you recorded in Part 4 of this experiment, calculate the **mass of CaCO3 extracted**. **(h) – (g)**	(l) _____ g $CaCO_3$
5. Calculate % NaCl recovered by dividing the mass of NaCl extracted **(j)** by the mass of your unknown sample mixture **(i)**. Express the fraction as a percentage (multiply by 100).	(m) _____ % NaCl
6. Calculate % sand recovered by dividing the mass of sand recovered **(k)** by the mass of your unknown sample mixture **(i)** Express the fraction as a percentage (multiply by 100).	(n) _____% sand
7. Calculate % $CaCO_3$ recovered by dividing the mass of $CaCO_3$ extracted **(l)** by the mass of your unknown sample mixture **(i).** Express the fraction as a percentage (multiply by 100).	(o) _____% $CaCO_3$
8. Calculate total mass of components recovered by adding the mass of recovered NaCl **(j)**, recovered sand **(k)** and recovered $CaCO_3$ **(l)**.	(p) _____ g NaCl + sand + $CaCO_3$ recovered
9. Calculate % recovery by dividing the total mass **(p)** by the mass of your unknown sample mixture **(i)**. Express the fraction as a percentage (multiply by 100).	(q) _____ % recovery

Post-Lab Questions:

1. In Part 3 Step 3, after decanting the supernatant into the beaker, the residue (sand) was washed a few times with laboratory water. Explain why this washing step is necessary.

2. Obtain the actual percentage of each component in your unknown mixture from your instructor. Compare the values and explain why your calculated percentages are higher or lower than the actual values. What may have caused the error?

3. Student Fumblefingers did the experiment but did not evaporate the NaCl to complete dryness. What is the effect on the following calculated values?

a) % NaCl

b) % recovery

4. The calcium carbonate was reacted with hydrochloric acid to separate it from the sand. A partial chemical equation for this reaction is listed below:

$CaCO_3(s) + 2\ HCl\ (_{aq}) \rightarrow Ca^{+2}(_{aq}) + 2\ Cl^{-}(_{aq}) + H_2O + something$

Note: The subscript "$(_s)$" means solid. The subscript "$(_{aq})$" means aqueous or dissolved in a solution.

a) What do you think "something" might be? Would the generation of "something" be consistent with your observations?

b) Now that you have an idea of what "something" is, write out the fully balanced chemical equation for the reaction in Part 4.

Notes:

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1.4: Experiment_604_Thermal_Decomposition_of_Sodium_Bicarbo

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 604: Thermal Decomposition of Sodium Bicarbonate**

Experiment 604: Thermal Decomposition of Sodium Bicarbonate

Section 1: Purpose and Summary

Determine if sodium bicarbonate decomposes by reaction A or reaction B when heated.

$NaHCO_3(s) \rightarrow Na_2CO_3(s)+H_2O(g)+CO_2(g)$								**Reaction A**
	Sodium bicarbonate		Sodium carbonate		Water vapor		Carbon dioxide	
$NaHCO_3(s) \rightarrow Na_2O(s)+H_2O(g)+CO_2(g)$								**Reaction B**
	sodium bicarbonate		Sodium oxide		Water vapor		Carbon dioxide	

$NaHCO_3$ is commonly called sodium bicarbonate. When $NaHCO_3$ is heated above 110 °C, H_2O and CO_2 are produced by a chemical change. After the decomposition reaction is complete, a white solid residue remains. Among all possible reactions, the two possibilities listed above are most likely to explain these observations.

Students are to choose between reactions (A) and (B) on the basis of their laboratory work, which will involve heating a weighed sample of sodium bicarbonate in a crucible and weighing the residue left in the crucible after heating. From the mass of the solid product ("residue"), students should be able to decide if it is Na_2CO_3 or Na_2O.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Wear your safety goggles.

Do not attempt to move a hot crucible; allow sufficient time for it to cool.

Use care when handling HCl solutions. If any HCl splashes on your skin or clothing, rinse it off immediately with plenty of water.

Waste Disposal:

The solid waste may be discarded in one of the regular garbage cans.

Section 3: Procedure

Part 1: Clean and Weigh Crucible

1. Obtain a clean, dry crucible that has no cracks.
2. Place the crucible in a clay triangle, on a ring attached to a ring stand. Make certain that the crucible hangs securely in the triangle; ask for help if it seems shaky. Put the lid on the crucible.
3. Heat the crucible and lid for 10 minutes with a high flame, until the bottom of the crucible glows red hot. Allow the crucible to cool in the clay triangle. (This step is to clean out the crucible.)
4. When they have reached room temperature, weigh the crucible and lid. Record the mass in the data table below.

Part 2: Measure Mass of Residue after Heating

1. Add sodium bicarbonate to the crucible until it is 3/4 full. Weigh the crucible, sodium bicarbonate, and lid. Record the mass in the data table below.
2. Obtain the mass of the sodium bicarbonate by subtraction. Show your work below and enter the mass of the sodium bicarbonate in the data table

Mass of $NaHCO_3$	=	*Mass of crucible, lid and $NaHCO_3$*	-	*Mass of crucible and lid*

3. Using a medium-sized flame, heat the sample for 15 minutes. Do NOT heat the sample until it is red hot. Cool it in place as before, and then weigh it. Record the mass of the crucible, lid and residue in the data table below.
4. Calculate the mass of the residue remaining by subtraction. Show your work below and enter the mass of the residue in the data table below.

Mass of residue	=	*Mass of crucible, lid and residue*	-	*Mass of crucible and lid*

5. Heat again, this time for 10 minutes, then cool and weigh as before. Record the mass of the crucible, lid and residue in the data table below.
6. Calculate the mass of the residue remaining by subtraction. Show your work below and enter the mass of the residue in the data table below.

7. Heat for 10 minutes for a third time, then cool and weigh as before. Record the mass of the crucible, lid and residue in the data table below.
8. Calculate the mass of the residue remaining by subtraction. Show your work below and enter the mass of the residue in the data table below.

Part 3: Data Table

	Before heating	After first heating	After second heating	After third heating
Mass of crucible, lid & sample (g)				
Mass of crucible & lid (g)				
Mass of sample (g)				

Part 4: Test for Presence of Carbonate

Carbonate salts such as Na_2CO_3 react with acids to form CO_2 gas. If the solid fizzes when some HCl solution is added to it, this indicates that the solid is a carbonate or bicarbonate. Fizzing will not be observed if the product is Na_2O. Add a few drops of 6 M hydrochloric acid to the solid product in your crucible to double-check your results. Record your observations.	Observations:

Section 4: Calculations

Part 1: Balance Chemical Equations

Before attempting the calculations, balance the chemical equations for reactions A and B below.	
___$NaHCO_3(s) \rightarrow$ ___$Na_2CO_3(s)$+___$H_2O(g)$+___$CO_2(g)$	**Reaction A**
Reactants Na atoms: _____ C atoms: _____ H atoms: _____ O atoms: _____	**Products** Na atoms: _____ C atoms: _____ H atoms: _____ O atoms: _____

___$NaHCO_3(s)$ → ___$Na_2O(s)$ + ___$H_2O(g)$ + ___$CO_2(g)$		**Reaction B**
Reactants Na atoms: _____ C atoms: _____ H atoms: _____ O atoms: _____	**Products** Na atoms: _____ C atoms: _____ H atoms: _____ O atoms: _____	

Part 2: Yield Calculations

1. Starting with the mass of $NaHCO_3$ used, calculate the theoretical yield of Na_2CO_3 if reaction A occurred. Show your work below.

$$____\ g\ NaHCO_3 \left(\frac{1\ mol\ NaHCO_3}{____\ g\ NaHCO_3} \right) = ____ mol\ NaHCO_3$$

$$____ molNaHCO_3 \left(\frac{____ mol\ Na_2CO_3}{____ mol\ NaHCO_3} \right) = ____ mol\ Na_2CO_3$$

$$____ mol\ Na_2CO_3 \left(\frac{____ g\ Na_2CO_3}{____ mol\ Na_2CO_3} \right) = ____\ g\ Na_2CO_3$$

Theoretical yield of Na_2CO_3:

_____

2. Starting with the mass of $NaHCO_3$ used, calculate the theoretical yield of Na_2O if reaction A occurred. Show your work below.

Theoretical yield of Na_2O:

_____

3. Calculate the percent yield of Na_2CO_3 assuming reaction A occurred. Show you work below.

% ____ yield= $\frac{\text{actual yield}}{\text{theoretical yield}} \times 100$

Percent yield of Na_2CO_3:

_____

Post Lab Questions:

1. Compare the observed mass of residue after heating to the predicted masses of Na_2CO_3 and Na_2O. Which reaction actually occurred and which product was formed?
2. Consider the percent yield calculated for Na_2CO_3 and the percent yield calculated for Na_2O. Do these numbers support your conclusion about which reaction is really occurring? How?
3. Did your observations during Part 4: Test for Presence of Carbonate agree with your conclusions based on the weight data? Explain?

Notes:

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1.5: Experiment_605_Hydrates_1_2_1

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 605: Hydrates**

Experiment 605: Hydrates

Section 1: Purpose and Summary

- Determine the percent water of hydration in a hydrate sample.
- Determine the number of moles of water, *x*, per mole of anhydrous salt and write the chemical formula of the hydrate sample.

Hydrates are ionic compounds that contain water molecules as part of their crystal structure. The bound water is called the water of hydration. Some compounds lose this water of hydration spontaneously (efflorescent) while some may require heating. On the other hand, an anhydrous salt (without water) can absorb water from the atmosphere and spontaneously dissolve in its own water of hydration (deliquescent).

A hydrate contains a definite number of water molecules bound to each ionic compound or *anhydrous salt*. In this experiment, students will dehydrate an unknown hydrate sample by heating, and calculate the amount of water lost in the process. Students will determine the ratio between the moles of water lost and the moles of anhydrous salt and write the chemical formula of the hydrate sample.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

A hot crucible will break if placed directly on a cold surface. Set hot crucibles on to wire screens to cool.

Waste Disposal:

When you are finished with the experiment, discard solid waste in the container marked ‘solid waste’ in the fume hood.

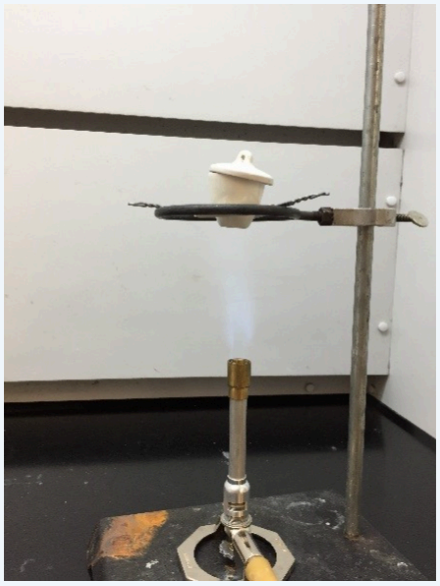
Section 3: Procedure

Part 1: Deliquescence and Efflorescence

Prepare two clean and dry watch glasses. Transfer a few crystals of sodium sulfate decahydrate, $Na_2SO_4{\times}10H_2O$, into one watch glass, and anhydrous calcium chloride, $CaCl_2$, into the other. Observe each sample occasionally as you perform the rest of this experiment. Record your observations below.	
$Na_2SO_4{\times}10H_2O$	**$CaCl_2$**

Part 2: Formula of an unknown hydrate

Possible unknowns: $CaCl_2 \times xH_2O$, $CaSO_4 \times xH_2O$, $AlK(SO_4)_2 \times xH_2O$, $MgSO_4 \times xH_2O$

1. Obtain an unknown hydrate from your instructor. Record identification code for your unknown.	Identification code for unknown:
2. Assemble a wire triangle supported on an iron ring attached to a ring stand. Put a clean, dry porcelain crucible and lid on the wire triangle and heat over a Bunsen burner flame for about three (3) minutes to ensure complete dryness. An example setup is shown:  Allow the crucible to cool on the wire triangle. When the crucible is cool and safe to touch, weigh on an analytical balance. Record exact mass of the crucible and lid.	Mass of crucible and lid: 1. _____ grams
3. Transfer 2 – 4 grams of your unknown sample into the crucible and weigh again. Record exact mass. NOTE: You need to have both the total mass of the crucible, lid and sample as well as just the sample. You can determine the mass of the sample by subtraction of the mass of the crucible (a).	Mass of crucible, lid and sample (before heating): 2. _____ grams
4. Put the crucible and sample back on the wire triangle. Position the crucible such that it is at a slight angle on the triangle. Place the crucible lid so that the lid is slightly ajar. Gently heat the crucible for 5 minutes over a Bunsen burner flame (slowly move the burner back and forth across the bottom of the crucible).	
5. After gentle heating for about 5 minutes, increase the heat applied to the crucible, lid, and sample until the bottom of the crucible turns red. Continue providing high heat for additional 10 minutes. Let the crucible cool for 5 to 10 minutes. Using crucible tongs, place the lid and the crucible on a wire gauze on the bench to finish cooling to room temperature.	

6. When the crucible is cool and safe to touch, weigh on an analytical balance. Record exact mass. Be sure to include the lid with the crucible on the balance.	Mass of crucible, lid and sample (after final heating): 3. _____ grams
7. To ensure complete dehydration of sample, reheat the crucible, lid, and sample as in step #5, except to heat it for 5 minutes. Cool, and weigh again. Record exact mass. (*Repeat this process, if necessary, until the mass of the sample is within 0.01 g of the previously recorded mass.*)	Mass of crucible, lid and sample (after 2nd, etc. final heating): 4. _____ grams

Part 3: Reversibility of hydration

1. Transfer a small amount of solid copper (II) sulfate pentahydrate, $CuSO_4 \times 5H_2O$, that just fills the bottom of a clean, dry 150-mm (medium size) test tube.
2. Using a test tube holder, grasp the test tube containing the hydrate and heat over a Bunsen burner flame while holding the test tube at a 45° angle.
3. While heating, closely observe the solid and the inside wall of the test tube. Record your observations.
4. When the solid residue seems to be completely dehydrated, allow the test tube to cool completely. Then, add a few drops of laboratory water to the solid in the test tube. What changes did you see? Record your observations.

Appearance of $CuSO_4.5H_2O$:

Appearance of solid after heating:

Appearance of the inside wall of the test tube after heating:

Appearance of solid residue after adding a few drops of laboratory water:

Section 4: Calculations

1. From the masses you recorded in Part 2 of this experiment, calculate the mass of the unknown hydrate sample. **(b) – (a)**	Mass of the unknown hydrate sample: (e) _____ grams

2. Calculate the mass of the heated (dehydrated) sample. This is also called the *anhydrous salt*. **(d) – (a)**	Mass of the heated sample (dehydrated): (f) _____ grams
3. From the mass of the hydrate and the mass of the heated sample, calculate the mass of water lost. **(e) – (f)**	Mass of water lost: (g) _____ grams
4. Calculate percent water in the hydrate. % water= $\frac{\text{mass of water lost (g)}}{\text{mass of hydrate (e)}}$ = x 100	***Percent water in the hydrate:***
5. Ask for the identity of the unknown hydrate sample from your instructor. Refer to a periodic table to obtain the molar mass of the anhydrous salt. Then, using this information and the mass of the heated sample, calculate the number of moles of the anhydrous salt. Identify of the anhydrous salt: _____ Molar mass of anhydrous salt: _____g/mol Show your equation here:	Moles of anhydrous salt: (h) _____ mol
6. Similarly, determine the molar mass of water. Then, calculate the number of moles of water lost from the sample. Molar mass of water, H_2O: _____g/mol Show your equation here:	Moles of water lost: (i) _____ mol
7. Calculate the ratio of moles of water lost to moles of anhydrous salt. **(i) ÷ (h)**	$\frac{\text{mole of } H_2O}{\text{mole of anhydrous salt}}$ ratio (j) ___ : ___
8. The ratio calculated **(j)** is expressed in the formula of the compound (hydrate). Write the chemical formula of the hydrated form of your unknown sample. *For example*, if a given amount of hydrated copper(II) sulfate gave off 0.060 mole of H_2O and left behind 0.012 mole of anhydrous copper(II) sulfate, $CuSO_4$, then the ratio of H_2O to $CuSO_4$ is 5:1, and the formula would be written as $CuSO_4 \times \mathbf{5}H_2O$.	**Chemical formula of the unknown sample:**

Post-lab Questions

1. Calculate the mass percent of water for the hydrate, $LiNO_3 \times 3H_2O$.
2. What will be the probable effect if you kept the crucible completely covered during the entire heating and cooling processes? Would your calculated percent water in the hydrate be high, low, or unaffected? Explain.
3. If 2.752 g sample of $Ca(NO_3)_2 \times xH_2O$ is heated to constant mass, the residue weighs 1.941 g. Determine the value of *x* and the formula of the hydrate.

Notes:

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1.6: Experiment_606_Double Displacement Reactions_1_2_3

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 606: Double Displacement Reactions**

Experiment 606: Double Displacement Reactions

Section 1: Purpose and Summary

- Carry out double displacement reactions involving different aqueous solutions.
- Identify reactions that form precipitates, evolve gases, and increase temperature after mixing.
- Predict whether a double displacement reaction will occur between two solutions.
- Write molecular, complete ionic, and net ionic equations.

In this experiment, students will combine two aqueous solutions and determine whether a reaction occurs. Based on solubility rules, students will predict whether a double displacement reaction will occur from a given set of aqueous solutions. The students will then carry out the reaction to confirm their prediction.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Waste Disposal:

While you are doing the experiment, collect your reaction mixtures into a waste beaker.

When you are finished with the experiment, pour the contents of the waste beaker (liquid waste only) into the inorganic waste container in the fume hood. Rinse water from the beaker does not need to be captured, just the bulk material.

Section 3: Procedure

Each reaction in this experiment involves mixing two aqueous solutions in a test tube. Clean, rinse with laboratory water, and dry fourteen (14) test tubes. Label each test tube with the reaction mixture. Set on a test tube rack.

To determine whether a reaction occurs or not, observe any bubble formation (gas evolution) in the solution, formation of a solid (precipitate), or increase in temperature of the reaction mixture. Bubble formation is due to the formation of a gaseous product. If the solution becomes cloudy, this is evidence for the formation of fine, suspended solid particles of the precipitate.

Part 1: Observing double displacement reactions

Obtain about 1 mL (approximately 20 drops) each of the indicated solutions in a test tube. Record your observations. Write the molecular, complete ionic, and net ionic equations for the reaction. If no observable change is noted, write ‘no reaction’ on the reactant side of the equation; no need to write the complete ionic and net ionic equations.

Do one reaction at a time to avoid mixing up of solutions. It is important not to mix up droppers (if used) to avoid contamination of reagents. This may also lead to ‘false positive’ observations.

NOTE: All solutions are at a concentration of **0.1 Molar** (moles per liter) unless otherwise indicated*.

1. **Sodium chloride (aq) + potassium nitrate (aq)**

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

2. **Sodium chloride (aq) + silver nitrate (aq)**

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

3. **Sodium carbonate (aq) + hydrochloric acid (aq)**

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

4. **Sodium hydroxide (aq) + hydrochloric acid (aq)**

NOTE: Add one drop of phenolphthalein indicator separately to the sodium hydroxide and hydrochloric acid before mixing. Mix in a specific sequence.

4.1. Transfer ½ of the sodium hydroxide to the hydrochloric acid. (Excess hydrochloric acid)

4.2. Transfer the rest of the sodium hydroxide to the hydrochloric acid. (Equal amounts)

4.3. Add an additional 1-2 mL of sodium hydroxide to the hydrochloric acid. (Excess sodium hydroxide)

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

5. **Barium chloride (aq) + sodium sulfate (aq)**

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

6. **Lead (II) nitrate (aq) + potassium iodide (aq)**

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

7. **Calcium chloride (aq) + sodium carbonate (aq)**

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

8. ***Saturated sodium chloride (aq) + lead (II) nitrate (aq)**

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

9. **Potassium sulfate (aq) + ammonium chloride (aq)**

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

10. **Sodium hydroxide (aq) + nitric acid (aq)**

NOTE: Add one drop of phenolphthalein indicator separately to the sodium hydroxide and nitric acid before mixing. Mix in a specific sequence.

10.1. Transfer ½ of the sodium hydroxide to the nitric acid. (Excess nitric acid)

10.2. Transfer the rest of the sodium hydroxide to the nitric acid. (Equal amounts)

10.3. Add an additional 1-2 ml of sodium hydroxide to the nitric acid. (Excess sodium hydroxide)

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

11. **Iron(III) chloride (aq) + *1 M sodium hydroxide (aq)**

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

12. **Zinc nitrate (aq) + *1 M sodium hydroxide (aq)**

Observations:

Molecular equation:

Complete ionic equation:

Net ionic equation:

Part 2: Predicting double displacement reactions

SOLUBILITY RULES for IONIC COMPOUNDS

1. Group 1 metal compounds (also called alkali metal compounds), acetates, nitrates, and ammonium compounds are all soluble.

2. Hydroxides of alkali metals and NH_4^{+1}, Ca^{+2}, Sr^{+2}, and Ba^{+2} are soluble. All others are insoluble.

3. All halides (chlorides etc.) are soluble except for those containing Ag^{+1}, Pb^{+2}, and Hg_2^{+2}.

4. Most sulfates are soluble, except for $BaSO_4$, $SrSO_4$, Ag_2SO_4, $PbSO_4$, and $CaSO_4$.

5. Most phosphates, carbonates, chromates and sulfides are insoluble (except those of the Group 1

metals and ammonium ion).

6. In addition, all acids (compounds that form an ionized H^+, HCl, H_2SO_4, HI, etc.) are soluble!

You have the following solutions available in the lab: NH_4Cl, NaOH, HCl, and $CuSO_4$.

1. Predict which pair of compounds will produce a precipitate. Use the solubility rules listed above.
2. Test your prediction by combining about 1 mL each of the two solutions in a clean and dry test tube. Record the results of your test below and write the net ionic equation for the reaction.
3. Compounds tested: Observations: Net ionic equation:
3. Predict which pair of compounds will produce a gaseous product (e.g. CO_2, NH_3). Before you test your prediction, wet a piece of red litmus paper and attach it to the bottom of a watch glass.
4. Test your prediction by combining about 1 mL each of the two solutions in a clean and dry test tube. Immediately cover the test tube with the watch glass with the red litmus paper. The red litmus paper will turn blue as evidence for the formation of gaseous ammonia in your reaction. Record the results of your test below and write the net ionic equation for the reaction.
Compounds tested: Observations: Net ionic equation:

Notes:

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1.7: Experiment_607_Single Displacement Reactions_1_1_1

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 607: Single Displacement Reactions**

Experiment 607: Single Displacement Reactions

Section 1: Purpose and Summary

- Carry out single displacement reactions involving (a) metals with salts of other metals, and (b) metals with dilute acid.
- Determine the order of activity of metals and hydrogen based on observations.

Note: 'Single displacement reactions' are sometimes called 'single replacement reactions'.

In this experiment, students will conduct different reactions between a metal and a salt of another metal. Students will also conduct reactions between metals and dilute acid solution. Based on observations, students will list the metals and hydrogen in order of activity.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Waste Disposal:

While you are doing the experiment, pour your liquid waste into a beaker. Separate the solid metal pieces from the waste solution.

Metal pieces should be rinsed with water, and then they may be disposed of in proper solid waste container.

When you are finished with the experiment, collect the liquids into a waste beaker (liquid waste only). Transfer the liquid waste into the inorganic waste container in the fume hood.

Section 3: Procedure

Some combinations of reactants used in this experiment may react slowly or be difficult to detect at times. **If you see no immediate visible result, set the test tube aside and allow it to stand for 5 to 10 minutes, then reexamine it.**

To determine whether a reaction occurs or not, observe any bubble formation (gas evolution) in the solution or a change in appearance of the metallic solid (note the color and/or texture).

Part 1: Reaction of metal with aqueous metal salt solution

1. Obtain four (4) pieces each of magnesium, copper, zinc, and iron metals. If necessary, clean the metal piece with fine sandpaper or steel wool until the surface is clean and shiny. Surface coatings on these metals may inhibit chemical reactions.

2. Rinse three (3) test tubes with laboratory water and shake out the excess water. Label them with Cu, Zn, and Fe. Set them up in your test tube rack.

3. In each of the test tubes, place about 1 mL (20 drops) of magnesium nitrate solution. Add a piece of metal (except for magnesium metal) to each test tube. Observe the metal pieces for evidence of reaction. Note, if any, change in the color of the solution. Record your observations in the table below. If no change is observed, write 'no reaction'.

4. Repeat steps 2 and 3, using a different metal salt solution and set of metals.

	with $Mg(NO_3)_2(aq)$
Cu(s)	
Zn(s)	
Fe(s)	

	with $Cu(NO_3)_2(aq)$
Mg(s)	
Zn(s)	
Fe(s)	

	with $Zn(NO_3)_2(aq)$
Mg(s)	
Cu(s)	
Fe(s)	

	with $Fe(NO_3)_3(aq)$
Mg(s)	
Cu(s)	
Zn(s)	

Part 2: Reaction of metal with dilute acid solution

1. Rinse four (4) test tubes with laboratory water and shake out the excess water. Label them with Mg, Cu, Zn, and Fe. Set them up in your test tube rack.
2. In each of the test tubes, place about 1 mL (20 drops) of hydrochloric acid solution. Add a piece of metal to each test tube. Observe the metal pieces and the solution for evidence of reaction. Record your observations in the table below. If no change is observed, write 'no reaction'.

	with HCl(aq)
Mg(s)	
Cu(s)	
Zn(s)	
Fe(s)	

Post-lab Questions:

1. Write balanced molecular equation, complete ionic equation and net ionic equation for each reaction that you observed in this experiment.
2. Based on your observations, list Mg, Cu, Zn, Fe, and H in order of increasing activity (least to highest).
3. Aluminum lies below magnesium and above zinc in the reactivity series of metals. Predict its reaction with each of the metal solutions and dilute acid solution used in this experiment.

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1.8: Experiment_608_Six Unknown Solutions_1_1_2

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Name	**Experiment 608: Six Unknown Solutions**

Experiment 608: Six Unknown Solutions

Section 1: Purpose and Summary

- Determine if a chemical reaction has taken place when two solutions are combined.
- Determine the identity of six unknown solutions.

In this experiment, students are provided with a set of six solutions: $Cu(NO_3)_2$, $CuSO_4$, $BaCl_2$, KIO_3, $Na_2C_2O_4$, and NaCl, and the same set of solutions in a set of 'unidentified' numbered/lettered bottles. Students will conduct different reactions that involve combination of two known solutions and generate experimental data that will help them identify the set of unknown solutions.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Wash your hands after working with metal solutions. Do not eat in the laboratory.

Waste Disposal:

While you are doing the experiment, collect all waste into a beaker.

When you are finished with the experiment, pour the contents of the waste beaker into the inorganic waste container in the fume hood.

Section 3: Procedure

To determine whether a reaction occurs or not, observe any bubble formation (gas evolution) in the solution, a change in color of the solution, or formation of a precipitate. Record your observations at the time of mixing. If there is a precipitate, write 'ppt', and indicate the color. Where there is no evidence of reaction, write 'NR'. See the suggested data tables after each section below.

Part 1: Known solutions

1. Obtain solutions containing the following compounds: $Cu(NO_3)_2$, $CuSO_4$, $BaCl_2$, KIO_3, Na_2C_2O4, and NaCl.
2. Obtain a clear plastic or white ceramic well plate and clean thoroughly with tap water. **Rinse well with laboratory water**. Shake off excess water to dry the plate.
3. Label the top and left side of your well plate with the compounds according to the data table.

4. In each of the wells, combine 2 to 3 drops of each pair of known solutions listed in the data table. Stir mixture with a toothpick or wooden splint. Record your observations.
5. Dispose of reaction mixtures into a designated waste beaker.
6. Clean your well plate thoroughly, rinse with laboratory water, and dry.

	NaCl	**KIO_3**	**$Cu(NO_3)2$**	**$Na_2C_2O_4$**	**$BaCl_2$**
$CuSO_4$					
$BaCl_2$					X
$Na_2C_2O_4$				X	X
$Cu(NO_3)_2$			X	X	X
KIO_3		X	X	X	X

Part 2: Unknown Solutions

1. Obtain a set of unknown solutions. **Record the number of the set you used.** This is very important! With no identification of which set you used, your work cannot be graded.

Record your Unknown Set # tested here: _____

2. Label the top and left side of your well plate with the compounds according to the data table.
3. In each of the wells, combine 2 to 3 drops of each pair of unknown solutions listed in the data table. Stir mixture with a toothpick or wooden splint. Record your observations.
4. Dispose of reaction mixtures into a designated waste beaker.
5. Clean your well plate thoroughly, rinse with laboratory water, and dry.

	F	E	D	C	B
A					
B					X
C				X	X
D			X	X	X
E		X	X	X	X

Section 4: Identification of Unknown Solutions

Based on your data and observations above, determine the identity of each of your unknown solutions. Clearly explain each step of your reasoning.

Solution	Identity	Experimental basis of your identification
A		
B		
C		
D		
E		
F		

Post-lab Questions:

Write the net ionic equation for each of the reactions that occurred in this experiment.

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1.9: Experiment_609_Determining the Molar Mass Using Ideal Gas Law_1_2_0

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 609: Molar Mass Determination Using Ideal Gas Law**

Experiment 609: Molar Mass Determination Using Ideal Gas Law

Section 1: Purpose and Summary

Determine the molar mass of an unknown volatile liquid by measuring the properties of its vapor phase after heating. Using the properties of its vapor phase, calculate its molar mass using the ideal gas law.

In this experiment, students will determine the molar mass of an unknown volatile liquid by measuring the properties of its vapor phase after heating. The liquid is heated until it is completely vaporized. The vapor phase is trapped in a flask and the properties of the vapor are measured. The molar mass of the sample is then calculated using the ideal gas law:

PV = nRT

where P is the pressure in atmospheres, V is the volume in liters, n is the number of moles of the gas, R is the gas constant, 0.0821 $\frac{\mathrm{L} \times \mathrm{atm}}{\mathrm{mol} \times \mathrm{K}} =$, and T is the Kelvin temperature.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Use tongs or mitts to handle hot flasks and beakers.

Waste Disposal:

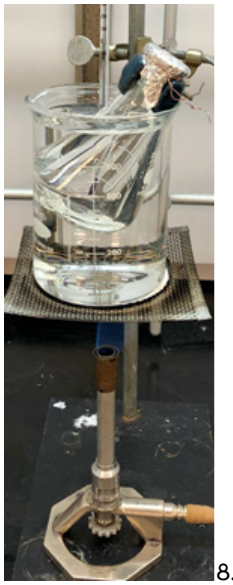
Discard any excess unknown liquids in the organic waste container in the fume hood.

Section 3: Procedure

Liquid sample in this experiment may be any of the following: acetone, isopropyl alcohol, ethyl acetate, or cyclopentane. Additional liquids may also be added at the request of the instructor.

1. Obtain a liquid sample from your instructor. Your instructor may choose to issue the liquid as an unknown sample.	Record the name of the liquid sample here, or the identification number of the unknown:

2. Obtain a clean, dry 125-mL Erlenmeyer flask, add one small crystal of dithizone (a dye) about the size of a grain of salt, and loosely cover it with a square piece of aluminum foil (about 5 cm x 5 cm). Secure the aluminum foil by tying a copper wire. It should look something like the following:  The crystal of dithizone is in the photo. It is very small. You do not need much dye. Weigh the flask, cover, dithizone crystal, and copper wire. Record mass to at least the nearest 1 milligram (0.001 grams).	Mass of Erlenmeyer flask with cover, dithizone and copper wire 1. _____ grams
3. Carefully remove the copper wire and aluminum foil cover and transfer about 5 mL of the liquid sample into the flask. **It is important to not lose any of the foil or copper wire.**	
4. Replace foil cover and crimp it tightly around the opening of the flask. Secure the foil with the copper wire. Make sure that there is no loose foil hanging down that may collect moisture from the water bath during the experiment.	
5. Use a pin to poke 1 small pinhole on the foil cover of the flask to allow the air and excess vapor to escape and maintain a pressure inside the flask that is equal to the atmospheric pressure.	
6. Prepare a water bath by filling a 600-mL beaker with about 400 mL of tap water. Add a few boiling chips. Place it on a wire gauze on an iron ring attached to a ring stand. Adjust the height of the ring to make sure that the hottest part of the flame of the Bunsen burner touches the bottom of the beaker. The beaker can be secured with an oversize ring to prevent it from falling over.	
7. Carefully attach a clamp to the neck of the flask and lower the flask, slightly tilted, as deep into the hot water bath as possible. Secure the clamp to the ring stand. One way to set up the apparatus is shown in the picture on the left. The thermometer is held in place with a buret clamp or a thermometer clamp. Other thermometers may be used as needed or available.	



8. Begin heating the water bath. Occasionally stir the water in the water bath with a stirring rod. Monitor the temperature of the water bath with a thermometer. Maintain the temperature of the water at around 95°C, or just below boiling throughout this experiment. Add additional water as needed to maintain the level of water.

The wire screen is necessary to disperse the heat from the flame to avoid breaking the beaker due to thermal shock.

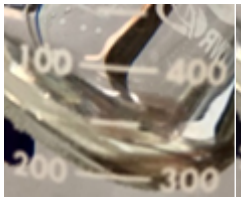
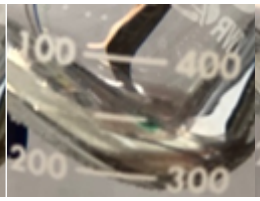
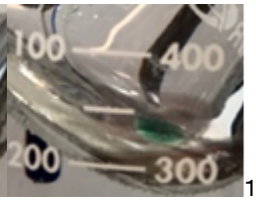
Alternatively, a different setup may be used with hot plates instead of Bunsen burners. Check with your instructor as to the specific procedure to use.

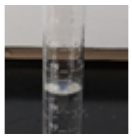
Use enough water to submerge a 125-ml Erlenmeyer flask but not enough to overflow the beaker. The thermometer is not shown.



9. Continue heating and watch the green color of the dithizone disappear – this indicates that the liquid sample has completely vaporized. If the water starts to boil while the liquid is evaporating, record this observation.

The dithizone dye is intensely green in solution, but dries to a dark solid residue.

  10. When you are sure that the liquid has completely vaporized, record the temperature of the water bath. Turn off the Bunsen burner. The pictures below show the close up of what the evaporating materials look like. Liquid present Almost gone Liquid gone	Temperature of the water bath 2. Trial 1: _____ °C Trial 2: _____ °C
11. Carefully remove the flask from the water bath by raising the clamp on the ring stand. The clamp may be hot; use thermal gloves or paper towels to protect from heat, if necessary. Keep the flask secured in the clamp until it has cooled to room temperature. Cooling may also be done faster by submerging the flask in an ice bath. While waiting for the flask to cool to room temperature, obtain the barometric pressure reading of the room and record it.	Barometric pressure (include units) 3. Trial 1: _____ Trial 2: _____
12. When the flask has cooled, wipe off traces of water on the outside of the flask and weigh the flask. The flask should contain some of the original liquid. This is a *condensed liquid*. Record exact mass of the flask, cover, copper wire and condensed liquid.	Mass of Erlenmeyer flask, cover, copper wire, dithizone, and condensed liquid 4. Trial 1: _____ grams Trial 2: _____ grams
13. **For a second trial**, add about 4-5 mL of the same liquid into the same flask. Replace cover and repeat steps 6 – 12. Use the same water bath. Since the water is already warm, the heating process should be faster.	

14. Determine the volume of the 125-mL Erlenmeyer flask. This is NOT the same as the "125-mL" written on the flask. To measure the volume, remove the copper wire and foil cover, rinse the flask with minimal acetone to remove the dithizone residue, and discard the first rinse into the organic waste container. Rinse the flask using tap water and then, fill it to the brim with tap water. Pour this water, without spilling, into a 250-mL graduated cylinder and record the volume to the nearest milliliter. It is best to use a single measurement for the volume of the water, but you can use a smaller graduated cylinder (100 ml or 50 ml) and measure the total volume in portions.   Fill the 125-mL Erlenmeyer flask full to the rim with water and measure the volume of the water. You need to determine the volume of the expanded vapor in the flask, not just where the volume markings are on the flask.	Volume of the Erlenmeyer flask = 5. _____ mL

Section 4. Calculations

	Trial 1	**Trial 2**
1. Convert your recorded barometric pressure **(c)** to atmospheres (atm). Assume that the pressure of the gas, **P**, is the same as the atmospheric pressure when the flask is full of the vapor of the unknown liquid. Show your equation here:	P = _____ atm	P = _____ atm
2. Convert the volume of the Erlenmeyer flask **(e)** to liters (l). Assume that the volume of the gas, **V**, is equal to the volume of the flask. Show your equation here:	V = _____ l	V = _____ l
3. Convert the temperature of water bath **(b)** to Kelvin. Assume that the temperature of the gas, **T**, is the same as the temperature of the water bath when the liquid has completely vaporized. Show your equation here:	T = _____ K	T = _____ K
4. Using the ideal gas law, PV = nRT, calculate the number of moles, **n**, of the gas. Show your equation here:	n = _____ mol	n = _____ mol

5. Calculate the mass of the sample: **(d) – (a)**	_____ g	_____ g
6. Using the mass and number of moles of the sample, calculate the molar mass of the liquid unknown. Show your equation here:	_____ g/mol	_____ g/mol
7. Calculate the average molar mass of the liquid unknown.	_____ g/mol	

Post Lab Questions:

1. Obtain the identity of your unknown liquid sample from your instructor. From the chemical formula of the compound, calculate its molar mass. Compare this value with the average molar mass you obtained from this experiment. Calculate the percent error.

2. There are some experimental errors or facts that could lead to a higher or lower calculated molar mass compared to the true value. In each case below, determine whether the experimental value would be higher or lower than the true value of the molar mass of the compound analyzed. Explain your answer briefly.

1. You took the flask out of the water bath before the entire sample had vaporized.

2. You took the flask out of the water bath before the entire sample had vaporized, and then put it back in the water bath to finish the vaporization process.

3. The temperature of the vapor inside the flask could be lower than the temperature of the water bath that you recorded.

4. You did not wipe off the traces of water outside the flask before weighing it.

3. When you calculated the moles of the gas (i.e. vapor inside the flask), you used the ideal gas law. However, the vapor is probably not acting like an 'ideal gas' under the conditions of this experiment. Explain why this is so. How would this behavior affect the value of the calculated molar mass? Would it be higher or smaller than the true value?

4. At the end of the experiment, you weighed your unknown sample in the flask after it has condensed back into a liquid.

1. Why can't you just measure the mass of the liquid that you originally put into the flask at the beginning of the experiment for use in the calculations?

2. How do we take into account the presence of air in the flask?

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1.10: Experiment_610_Line Emission Spectra and Flame Tests_1_1_3

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 610: Line Emission Spectra and Flame Tests**

Experiment 610: Flame Tests and Line Emission Spectra

Section 1: Purpose and Summary

- Observe the color of light emitted when a sample of a salt of a metal cation is heated
- Use the characteristic color of flames to identify ions in various laboratory samples
- Observe the emission spectra of various elements in gas discharge tubes

In the atomic structure of an element, electrons occupy shells and orbitals that correspond to a specific amount of energy. When energy is absorbed by the atom, these electrons are excited to higher energy shells or orbitals. Electrons in the excited state are unstable and will spontaneously go back to lower energy level, giving off light in the process. The wavelengths of light given off correspond to the energies involved when electrons move from one energy level to another. These electron movements or transitions show as lines in an emission spectrum for the element.

The flame test is a qualitative tool to help determine the identity of a metal ion in a compound. A solution containing metal ions is heated in a Bunsen burner flame where a characteristic color is given off that is visible to the naked eye. Heat excites the electrons in these metal ions to higher energy levels. When the electrons relax back to its original state, the same amount of energy is released and corresponds to the wavelength of light in the electromagnetic spectrum.

In this experiment, students will heat metal ion solutions and observe the color of the flame upon heating and use this information to identify ions present in a laboratory sample. Students will also observe the emission spectra of elements in gas discharge tubes.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Use caution when handling hydrochloric acid (HCl).

Waste Disposal:

Metal ion solutions should be disposed of in the Inorganic Hazardous Waste container.

Section 3: Procedure

Part 1: Flame tests

Different solutions of metal ions (such as K^{+}, Ba^{2+}, $^{Ca2+}$, Cu^{2+}, Sr^{2+}, and Na^{+}) may be used in this

experiment. Ask your instructor which ones you will be testing.

Note: Na^+ ion exhibits a particularly strong and persistent color. This ion is present as an impurity in most solutions since glass bottles contain Na^+ ions that can leach out. The strong color of Na^+ in the flame can mask other colors such as the pale lavender color of K^+. It is a good idea to test the Na^+ solution last.

1. Obtain a piece of nickel/chrome wire 10 – 15 cm long. Bend one end of the wire into the smallest possible loop, about 1 mm in diameter. This loop will hold a drop of solution when you are doing the flame test.
2. Obtain a few milliliters of 6 M HCl solution in a medium-size test tube. This will be used to clean the nickel/chrome wire.
3. Fill two additional medium sized test tubes with laboratory water. Label them “Rinse #1” and “Rinse #2”.
4. Cross contamination is a major issue for flame tests. Clean the nickel/chrome wire by first dipping into the 6 M HCl solution and then holding it in the hottest part of the Bunsen burner flame.
5. Dip the wire in “Rinse #1” and then hold it in the Bunsen burner flame. Repeat this with “Rinse #2”. Continue this process until there is no significant color to the flame that comes off the wire. Return to the 6 M HCl solution if needed. This step will be done between testing of each metal ion solution.
6. Obtain a sample of known metal ion solutions from your instructor into clean small test tubes. Only a few drops of each solution are needed.
7. Using a dropper, let a drop of the metal ion solution fall onto the loop of the wire. Do not let the dropper itself touch the wire as this can cause contamination of the solution.
8. Hold the wire in the hottest part of the Bunsen burner flame and observe the color that you see. Record your observation in the data table below.
9. Clean the wire by repeating steps #4-5. Repeat for all known solutions.
10. Obtain an unknown solution from your instructor. Each student should test his or her own unknown. You and your lab partner must use different unknowns.
11. Record the identification code of your unknown. Repeat steps 3 to 5 to test the unknown solution. Report the color of the flame you observed and determine the identity of the metal ion.

Metal Ion	**Observation/Flame Color**
K^+	

Ba^{2+}	
Ca^{2+}	
Cu^{2+}	
Sr^{2+}	
Na^{+}	

Identification Code of Unknown	**Observation/Flame Color**	**Identity of the Metal Ion**

Part 2: Spectroscopic Examination

A flame test will give a characteristic color which is the sum of all specific emissions for that element. The color from the flame test can be separated into its individual components using a prism, diffraction grating, or reflection grating. The following procedure uses a diffraction grating, but may be adapted with spectroscopes or prisms as appropriate.

As seen from the flame tests, the color of the metal ion tends to be brief, which makes it difficult to observe. Gas discharge tubes can be used to show a continuous set of colors from the specific element of interest. This makes an emission spectrum easier to observe.

1. Look through a diffraction grating at daylight or a regular tungsten light bulb. All of these sources give "continuous" rather than "discrete line" spectra; that is, they give the whole rainbow of colors, each merging into the next.
2. Darken the area and use the diffraction grating to look at the displayed elements in gas discharge tubes. Record the spectrum produced by each. Use crayons or colored pencils to record the colors seen by making vertical lines on the space below to represent the spectra obtained.

Spectra #1 Element in gas discharge tube: _____

Spectra #2 Element in gas discharge tube: _____

Spectra #3 Element in gas discharge tube: _____

Spectra #4 Element in gas discharge tube: _____

Spectra #5 Element in gas discharge tube: _____

Post Lab Questions:

To help you answer the following questions, refer to your textbook and your lecture notes.

1. Fill in the following table regarding the hydrogen spectrum. Use units of electron volts (eV) where 1 eV = 1.60218 x 10^{-19} Joules. Refer to your textbook for other unit conversions.

Color	Known λ(nm)	E (in eV)
Red	656.4	
Turquoise	486.3	
Purple	434.2	

2. Show the calculation for energy, *E*, of any two of the above colors using the following equation:

$E = \frac{hc}{\lambda}$

3. Show the calculation for the conversion of one of the above energies to the corresponding photon energy in units of kJ/mole. (Hint: *E* in the above equations is the energy per photon.).

4. What causes light to be emitted by an atom?

5. Why are only certain wavelengths emitted from an atom? (In other words, why do the spectra show lines of light instead of a continuous rainbow of light?)

6. Why does hydrogen emit different wavelengths of light than mercury?

Notes:

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1.11: Experiment_611_Periodic Properties_2_0

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 611: Periodic Properties**

Experiment 611: Periodic Properties

Section 1: Purpose and Summary

- Carry out reactions involving Group 2 cations and Group 17 halides.
- Identify an unknown solution based on the properties observed for the ions.

NOTE: Some older textbooks use prior group designations of Group 2A and Group 7A. We will be using the current designations of Group 2 (Be, Mg, Ca, Sr, Ba, Ra) and Group 17 (F, Cl, Br, I, At). Note that Be, Ra and At are not included in this experiment.

In this experiment, students will conduct a series of reactions involving some alkaline earth metal ions and halide ions. Students will determine observable trends and characteristic reactions within each group. Using the information gathered, students will determine the elements present in an unknown solution.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Silver nitrate ($AgNO_3$) solutions will react with skin and leave brown stains. Avoid direct contact with silver nitrate solutions. Contact may not be seen until after exposure to direct sunlight.

Sodium fluoride solutions will react with glass and should not have long term contact with glass (short time periods with dilute solutions are not generally a problem).

Waste Disposal:

While you are doing the experiment, collect your reaction mixture into a waste beaker.

When you are finished with the experiment, pour the contents of the waste beaker into the inorganic waste container in the fume hood.

Section 3: Procedure

Part 1: Reactions involving Group 2 cations

Transfer about 5 mL each of $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$, and $Ba(NO_3)_2$ solutions into four (4) labeled large test tubes. The NO_3- ion is a spectator ion in the solution; hence, will not interfere with any of the reactions.

Label four (4) small test tubes with Mg^{2+}, $Ca2^{+}$, Sr^{2+}, and Ba^{2+} for use in each test reaction below. The test tubes must be cleaned and rinsed with laboratory water between tests. They do not have to be dried.

Note: All reactions below are double displacement reactions. Observe whether or not precipitate forms. If no reaction is observed, write ‘no reaction’.

1. **Reaction with H_2SO_4**

1. Add about 10 drops of each of the metal ion solutions into the labeled small test tubes.
2. To each test tube, add about 10 drops of 1 M H_2SO_4. Mix the contents thoroughly.
3. Record your observations in the data table below, including the color of the precipitate formed.
4. Discard reaction mixtures in a designated waste beaker.

2. **Reaction with NaOH**

1. Add about 10 drops each of the metal ion solutions into the labeled small test tubes.
2. To each test tube, add about 10 drops of 1 M NaOH. Mix the contents thoroughly.
3. Record your observations in the data table below, including the color of the precipitate formed.
4. Discard reaction mixtures in a designated waste beaker.

3. **Reaction with $(NH_4)_2C_2O_4$**

(a) Add about 10 drops each of the metal ion solutions into the labeled small test tubes.

(b) To each test tube, add about 10 drops of 0.25 M $(NH_4)_2C_2O_4$. Mix the contents thoroughly.

(c) Record your observations in the data table below, including the color of the precipitate formed.

(d) Discard reaction mixtures in a designated waste beaker.

4. **Reaction with $NaBrO_3$**

(a) Add about 10 drops each of the metal ion solutions into the labeled small test tubes.

(b) To each test tube, add about 10 drops of 1 M $NaBrO_3$. Mix the contents thoroughly.

(c) Record your observations in the data table below, including the color of the precipitate formed.

(d) Discard reaction mixtures in a designated waste beaker.

	H_2SO_4	NaOH	$(NH_4)_2C_2O_4$	
Mg^{2+}				
Ca^{2+}				

Sr^{2+}				
Ba^{2+}				

Part 2. Reactions involving Group 17 halides

Transfer about 1 mL each of NaF, NaCl, NaBr, and NaI solutions into four (4) labeled test tubes. These solutions should all be at 0.1 M for concentration. The Na^+ ion is a spectator ion in the solution; hence, will not interfere with any of the reactions.

Label four (4) small test tubes with F^-, Cl^-, Br^- and I^- for use in each test reaction below. The test tubes must be cleaned and rinsed with laboratory water between tests. They do not have to be dried.

1. **Reaction with $AgNO_3$**

The reaction of the halides with silver ion is a precipitation reaction. Observe whether or not precipitate forms. Cloudiness is due to the formation of fine suspended solid particles in solution. If no reaction is observed, write 'no reaction'.

1. Add about 10 drops of each of the halide solutions into the labeled small test tubes.
2. To each test tube, add about 5 drops of 0.1 M $AgNO_3$. Mix the contents thoroughly.
3. Record your observations in the data table below, including the colors of precipitates formed.
4. Discard reaction mixtures in a designated waste beaker.

2. **Reaction with Cl_2× H_2O**

The reaction with chlorine water with a halide ion is an example of a redox reaction. Chlorine removes an electron from the halide ion and converts these ions to the diatomic halogens. The halide ions are colorless in solution. Bromine water and iodine water, on the other hand, are colored in solution. Observe the color changes in the following reaction.

1. Add a few drops of each of the four halide ion solutions to the labeled small test tubes.
2. Add about 1 mL of chlorine water to each test tube. Mix the contents thoroughly.
3. Record your observations in the data table below, noting the distinct color of the solutions.
4. Discard reaction mixtures in a designated waste beaker.

	$AgNO_3$	Cl_2× H_2O
F^-		
Cl^-		

Br^-		
I^-		

Part 3: Identification of Unknown

Obtain an unknown solution from your instructor and record the identification code. This unknown solution is a solution of a **Group 2 halide or a nitrate compound** in water (for example, $MgCl_2$, $CaBr_2$, etc.). Perform each of the tests done in Parts 1 and 2, record your observations in the data table below. Based on these observations, identify the ions that may be present in your unknown solution, then write its chemical formula and name.

Test Reaction	**Observation**	
Group 2 cation		
(a) with H_2SO_4		
(b) with NaOH		
(c) with $(NH_4)_2C_2O_4$		
(d) with $NaBrO_3$		
Group 17 halide		
(a) with $AgNO_3$		
(b) with $Cl_2 \times H_2O$		
Identification code of unknown		
Identity of the unknown	**Chemical Formula Name**	

Post-lab Question:

Write the net ionic equation for each reaction that occurred in this experiment. There is no need to write an equation for reaction mixtures recorded with 'no reaction'.

Notes:

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1.12: Experiment_612_Beer's Law_1_3_4

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 612: Beer's Law**

Experiment 612: Beer's Law

Section 1: Purpose and Summary

Determine the concentration of copper(II) ions in solution using spectrophotometric analysis.

Many substances absorb UV (ultraviolet) or visible light. When a substance absorbs certain frequencies (colors) of light, some of them are transmitted, and the solution appears colored. The color you see is the opposite of the color it absorbs in the basic color wheel (ROYGBV). For example, copper(II) sulfate solution is blue because the Cu^{2+} ion absorbs visible light in the 600 – 650 nm range (orange).

A spectrophotometer is an instrument used to measure the amount of light absorbed by a sample. A light source directs light into the sample and the amount of light absorbed is measured as either absorbance or % transmittance. If all the light passes through the sample without any absorption, then the absorbance reading on the spectrophotometer would be 'zero', and % transmittance is 100. The sample appears colorless.

In colored samples, the absorbance is related to the molar concentration of the absorbing substance in the solution. This relationship is known as **Beer's law** and is given by the equation: $\boldsymbol{A = abC}$, where *A* is the absorbance of the solution, *a* is the molar absorptivity of the substance, *b* is the path length of light passing through the solution, and *C* is the concentration of the solution in molarity (moles/L). If *a* and *b* are constant, then the absorbance, A, is directly proportional to the concentration, *C*. A solution of higher concentration absorbs more light, while a solution of lower concentration absorbs less light. If the absorbance of different solutions of known concentration is measured, a graph of absorbance vs. concentration results in a straight line.

In this experiment, students will prepare several dilute solutions from a standard stock solution. Using a spectrophotometer, students will measure the absorbance of each of the dilute solutions and construct a graph of absorbance vs concentration. Students will be given copper(II) sulfate of unknown concentration and measure its absorbance. Using this absorbance and the graph, students will determine the concentration of the unknown copper solution.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Spectrophotometers and cuvettes are expensive. Use them carefully.

Waste Disposal:

Copper(II) solutions should be disposed of in the Inorganic Hazardous Waste container. Dissolved copper salts are very toxic to marine and aquatic life.

Section 3: Procedure

Part 1: Preparation of dilute Cu^{2+} solutions

You will be preparing four dilute Cu^{2+} solutions from a 0.35 M standard Cu^{2+} solution.

1. For this part of the experiment, gather the following glassware and materials.

- Four (4) clean, and dry medium (150 mm) test tubes - labeled 1 through 4.
- Two (2) 100-mL beakers – one labeled 'water' and the other labeled 'Cu^{2+}'
- Large beaker – labeled 'waste'
- Two (2) 10-mL graduated pipets – one labeled 'water' and the other labeled 'Cu^{2+}'
- Pipet bulb

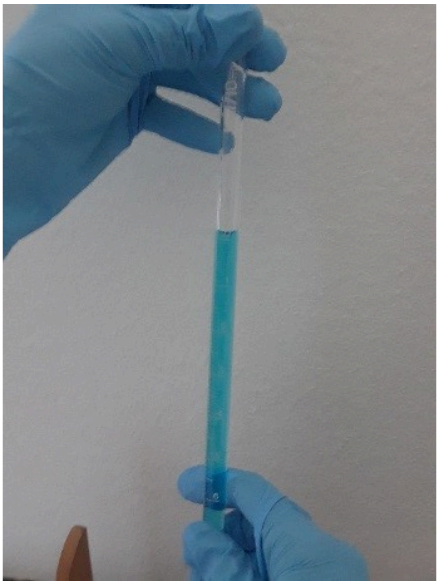
2. Rinse the beaker labeled 'Cu^{2+}' with a small amount of the standard Cu^{2+} solution and dump the rinse into the 'waste' beaker. Fill this beaker, about half-full, of standard Cu^{2+} solution. Record the exact concentration of the standard Cu^{2+} solution on the data table below.

3. Rinse the beaker labeled 'water' with a small amount of laboratory water and dump the rinse into the sink. Fill this beaker, about half-full, of laboratory water.

4. Rinse each labeled graduated pipet three times with the substance/solution it will be used for. Each time dump the rinse into the 'waste' beaker. Be careful not to get any water or Cu^{2+} solution sucked into the pipet bulb.

5. Now, you are ready to prepare the four dilute Cu^{2+} solutions. Each of the four test tubes will be filled with laboratory water and standard Cu^{2+} solutions with the amounts listed in the table below. Use your graduated pipets to withdraw the specified volumes. Make sure to record the actual volumes to two (2) decimal places. For example, if you must withdraw 2 mL of water, the water level can go beyond the 2 mL mark but record the exact volume, such as 2.01 mL.

To use the graduated pipet, first use the bulb to draw solution into the pipet above the zero mark. Cap the end of the pipet with your finger. Slowly allow air into the top of the pipet until the meniscus is just touching the '0.00' calibration mark. Move the end of the pipet over to your receiving test tube. Drain the pipet until the meniscus is just touching the '2.00' mark. Remove the pipet and return the additional liquid back to the stock you have in your beaker. 2.00 mL has now been transferred. Adjust these instructions for other volumes as appropriate.







6. Cover and securely close each test tube with a small piece of parafilm. Shake each solution well.

Test tube #	Volume (mL) standard Cu^{2+}	Volume (mL) laboratory water
1	2	8
2	4	6
3	6	4
4	8	2

Part 2. Absorbance Measurements

Follow instructions on the preparation and proper use of a spectrophotometer (Spectronic 20/200) (see Technique I: Use of Spectrophotometer of the laboratory manual for additional information). Turn on the spectrophotometer and allow it to warm up for 15 minutes. Set the wavelength to 620 nm. Absorbance measurements should all be done on the same day, using the same spectrophotometer.

1. Using a wash bottle filled with laboratory water, rinse and then fill (about ¾ full) a cuvette with laboratory water. This is your 'blank' sample. Wipe the outside wall of cuvette with Kimwipe™ and place it on the sample compartment in the spectrophotometer. Cover the sample compartment. Adjust spectrophotometer to 'zero' absorbance.
2. Discard the laboratory water from the cuvette into the sink. You are now ready to measure the absorbance of the four dilute Cu^{2+} solutions.
3. Rinse the cuvette three times with small portions of the dilute solutions you prepared in Part 2 of this experiment starting with test tube #1. Discard the rinse into the 'waste' beaker. Fill the cuvette with the solution (about ¾ full), wipe off the outside wall of the cuvette with Kimwipe™, place in the sample compartment, cover, and read the absorbance of the solution.
4. Discard the solution in the waste beaker.
5. Repeat steps 3 and 4 for all other dilute Cu^{2+} solutions. Record your data on the table below.

Concentration of standard Cu^{2+} solution (mol/L): _____

Test tube #	Actual Volume (mL) of standard Cu^{2+} (V_1)	Actual Volume (mL) of laboratory water	Total Volume (mL) of solution (V_2)	Absorbance	Concentration (M_2)*

1					
2					
3					
4					

***see Section 4. Calculations**

Part 3. Analysis of Unknown

1. Obtain an unknown sample from your instructor. Record its identification code.	**Identification code for unknown:**
2. Repeat steps 1 – 3 of Part 2 above and record the absorbance of your unknown sample solution.	**Absorbance of unknown Cu^{2+} solution:**
3. Discard the solution into the 'waste' beaker.	

Section 4: Calculations

SHOW YOUR WORK on the space provided below.

1. Refer to a periodic table and calculate the molar mass of $CuSO_4 \times 5H_2O$. Using this information, the mass of the solid, and the total volume of solution you made in Part 1, calculate the concentration (M) of the standard Cu^{2+} solution. Molar mass of $CuSO_4 \times 5H_2O$: _____ g/mol Show your equation here:	**Concentration of standard Cu^{2+} solution (M_2):** _____ mol/L
2. Using the dilution equation, $M_1V_1 = M_2V_2$, calculate the concentration of Cu^{2+} (M_2) in each of the diluted solutions in Part 2. Record the values on the last column of table in Part 3. Show one sample calculation here:	
3. After completing the table in Part 3, make a graph of absorbance (*y*-axis) vs. the concentration of Cu^{2+}, M_2 (*x*-axis). Use a graphing paper. Including a point at the origin (0,0), draw the best straight line among the points. An example is included at the end of this procedure.	

4. Using the straight line on the graph you made, determine the concentration of Cu^{2+} in your unknown solution from its absorbance. Estimate the value from the graph.	**Concentration of Cu^{2+} in your unknown sample:** _____ mol/L
5. Attach your graph to this lab report.	

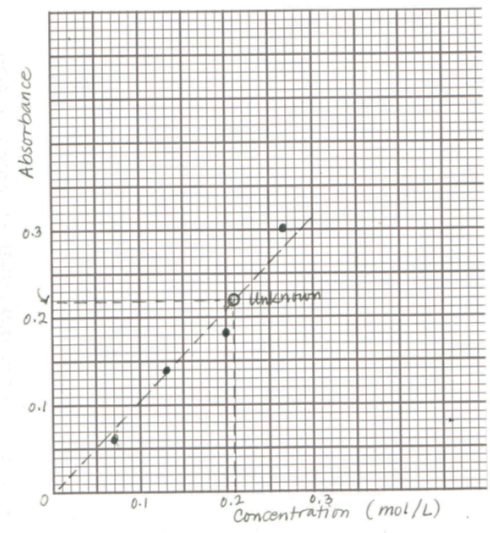
Post-Lab Questions:

1. Why is it necessary to rinse the pipets with the solution to be used in them before using the pipets?
2. Why is it necessary to rinse out the cuvette with the solution to be used in it before making our measurements?
3. Why must we use the same cuvette for all measurements?
4. What is the purpose of "zeroing" the spectrophotometer using a "blank" solution (water)?
5. A 6.000 g sample mixture containing copper was dissolved in 100.0 mL solution. The absorbance of this solution was 0.477 at 620 nm. Use your Beer's law graph to determine the concentration of copper ions in this solution. Then determine the mass percent copper in the sample mixture. Assume that the other components in the mixture did not interfere in the absorbance reading.

Example graph of data. The solid data points are from the measurements. The absorbance of an unknown solution is plotted on the graph with a hollow data point. The concentration of the unknown can be determined graphically from the calibration line by drawing a vertical line to the x-axis.

Note: This analysis may also be done by calculation.

Measured absorbance vs. Copper(II) Sulfate Concentration



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1.13: Experiment_613_Spectrophotometric Determination of Aspirin_1_2_2

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 613: Spectrophotometric Determination of Aspirin**

Experiment 613 Spectrophotometric Determination of Aspirin

Section 1: Purpose and Summary

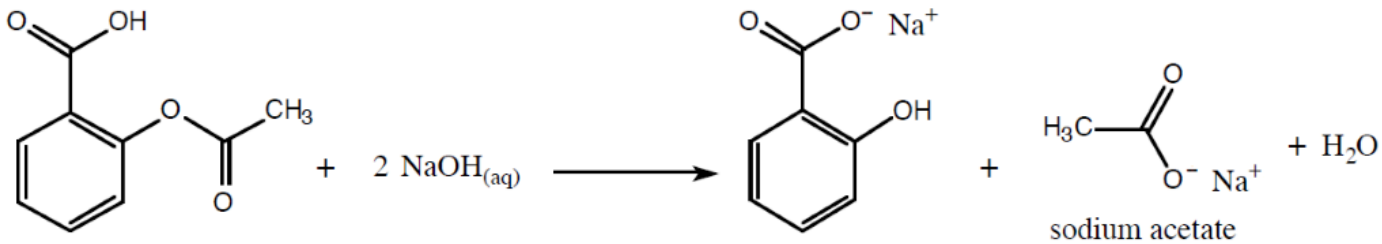
Most aspirin tablets are said to contain 5 grains of the active ingredient acetylsalicylic acid. (The grain is a unit of mass and is equal to 0.0647989 grams.) How reliable is this figure? This experiment will enable you to check up on the manufacturer.

The method of analysis that we will use is called "spectrometry" or "spectrophotometry". It depends on the fact that molecules can absorb electromagnetic radiation of certain wavelengths, using the energy of the radiation to "excite" electrons in their atoms. (Thus they have absorption spectra, just as isolated gas atoms do.) The greater the concentration of a particular molecule present in a sample, the more light of a particular wavelength the sample will be able to absorb. The absorbance of light by the

sample increases in direct proportion to the concentration of the molecule present. We will use electromagnetic radiation ("light") of wavelength (λ) 297 nm, which falls in the ultraviolet range. In this experiment, 297 nm light shines through the sample, and the amount of light absorbed by the sample is measured.

The chemical name of the active ingredient in aspirin is acetylsalicylic acid. In order to dissolve aspirin completely in water, you will convert it chemically to the salt sodium salicylate before measuring the absorbance. Notice that 1.00 mole of sodium salicylate is produced for every 1.00 mole of

acetylsalicylic acid (aspirin) used up:



acetylsalicylic acid (aspirin) sodium salicylate sodium acetate

Sodium salicylate will then be reacted with acidic Fe^{3+} to form salicylatoiron(III) complex, $[FeSal]^{+}$. This complex displays a maximum absorption at a wavelength of 525 nm and has a purplish red color. The absorption of the $[FeSal]^{+}$ complex will be directly proportional to the concentration of salicylate in the sample, but it will not tell us the actual concentration in any given sample. In order to be able to convert an instrument reading to an actual concentration of salicylate, we must first calibrate the instrument using solutions of known sodium salicylate concentration.

Overall, this experimental project consists of:

- Carefully preparing 5 solutions of **known** concentrations
- Measuring the absorbance of each **known** and **unknown** solution

- Carefully preparing an aspirin solution (of **unknown** concentration)
- (After lab) Preparing a calibration curve from the data of the 5 solutions of **known** concentrations
- (After lab) Using the calibration curve to calculate the amount of aspirin in one tablet or pill.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

All of the materials in this experiment are relatively harmless. Aspirin obtained in lab should not be ingested. Sodium hydroxide solutions are corrosive. Use of eye protection is recommended for all experimental procedures.

Waste Disposal:

Dispose of solutions down the sink drain with plenty of water. Dispose in the trash any filter paper and undissolved residue from the aspirin tablet.

Section 3: Procedure

Part 1: Preparing solutions of known concentrations

1. Ask your instructor to describe and demonstrate the use of volumetric pipets, volumetric flasks, and burets. 2. Into your smallest beaker, obtain 10-15 mL of the standard solution of approximately 0.03 M sodium salicylate. Record the actual concentration (include all the significant figures).	Concentration (Molarity) of the Standard Solution of sodium salicylate:
3. Using a rubber bulb and a 5.00 mL volumetric pipet, carefully measure 5.00 mL of the standard solution and place it in a 100.0 mL volumetric flask. Carefully fill the flask with laboratory water exactly to the 100 mL mark. Mix the sample thoroughly by carefully inverting the stoppered flask repeatedly for at least 2 minutes. (Hold the glass or rubber stopper tightly in place with your thumb. Make sure no liquid leaks out before the solution is completely mixed.) This is your **Stock Solution**.	
4. Obtain 6 clean and dry beakers that are larger than 100 mL. Label these beakers “Stock”, “1”, “2”, “3”, “4”, and “5”.	
5. Empty the 100.0 mL volumetric flask into the beaker labeled “Stock”. Rinse the volumetric flask with plenty of laboratory water.	
6. Fill a 50 mL buret using your Stock Solution. Fill it close to the 0.00 mL mark, making sure that you can accurately read the volume at the meniscus (the lowest part of the surface of the solution inside the buret). It is OK if the volume is not exactly 0.00 mL.	Initial Buret Reading (mL): (Include 2 decimal places!)
7. Now prepare the first known solution, **Solution #1**. Place the 100 mL volumetric flask below the buret, then turn the stopcock of the buret so that about **5 mL** drains into the volumetric flask. Afterwards, read the buret.	Buret Reading #1 (mL): (Include 2 decimal places!)

8. Quick check: Subtract the **Initial Buret Reading** from **Buret Reading #1**. This number should be close to 5 mL. Later you will use this volume to calculate the concentration (molarity) of sodium salicylate in Solution #1. Let's name this volume "Transfer Volume #1"	Calculate the difference between the Initial Buret Reading and Buret Reading #1. (Include 2 decimal places!) "Transfer Volume #1": _____ mL
9. To the 5 mL contained in the volumetric flask, carefully fill the flask with 0.010 M $FeCl_3$ in 0.1 M HCl solution the 100 mL mark and mix well. This is **Solution #1**. Empty the 100 mL volumetric flask into the beaker labeled "1". Rinse the volumetric flask with plenty of laboratory water.	
10. Now prepare the second known solution, **Solution #2**. Place the 100 mL volumetric flask below the buret, then turn the stopcock of the buret so that about **10 mL** drains into the volumetric flask. Afterwards, read the buret.	Buret Reading #2 (mL): (Include 2 decimal places!)
11. Subtract **Buret Reading #1** from **Buret Reading #2**. This number should be close to 10 mL. Record **Transfer Volume #2.** Carefully fill the volumetric flask with 0.010 M $FeCl_3$ in 0.1 M HCl solution exactly to the 100 mL mark and mix well. This is **Solution #2**. Empty the 100 mL volumetric flask into the beaker labeled "2". Rinse the volumetric flask with plenty of laboratory water.	Calculate the difference between Buret Readings #1 and #2. (Include 2 decimal places!) "Transfer Volume #2": _____ mL
12. Now prepare the third known solution, **Solution #3**. Place the 100 mL volumetric flask below the buret, then turn the stopcock of the buret so that about **15 mL** drains into the volumetric flask. Afterwards, read the buret.	Buret Reading #3 (mL): (Include 2 decimal places!)
13. Subtract **Buret Reading #2** from **Buret Reading #3**. This number should be close to 15 mL. Record **Transfer Volume #3.** Carefully fill the volumetric flask with 0.010 M $FeCl_3$ in 0.1 M HCl solution exactly to the 100 mL mark and mix well. This is **Solution #3**. Empty the 100 mL volumetric flask into the beaker labeled "3". Rinse the volumetric flask with plenty of laboratory water.	Calculate the difference between Buret Readings #2 and #3. (Include 2 decimal places!) "Transfer Volume #3": _____ mL
14. Refill the buret with your stock solution (use the beaker labeled "Stock"). Read the buret and record this volume (it should be close to the 0.00 mL mark). It is OK if the volume is not exactly 0.00 mL.	Initial Buret Reading After REFILL (mL): (Include 2 decimal places!)

15. Now prepare the fourth known solution, **Solution #4**. Place the 100 mL volumetric flask below the buret, then turn the stopcock of the buret so that about **20 mL** drains into the volumetric flask. Afterwards, read the buret.	Buret Reading #4 (mL): (Include 2 decimal places!)
16. Subtract the **Initial Buret Reading After REFILL** from **Buret Reading #4**. This number should be close to 20 mL. Record **Transfer Volume #4.** Carefully fill the volumetric flask with 0.010 M $FeCl_3$ in 0.1 M HCl solution exactly to the 100 mL mark and mix well. This is **Solution #4**. Empty the 100 mL volumetric flask into the beaker labeled "4". Rinse the volumetric flask with plenty of laboratory water.	Calculate the difference between Buret Reading #4 and the Initial Buret Reading After REFILL. (Include 2 decimal places!) "Transfer Volume #4": _____ mL
17. Finally, prepare the fifth known solution, **Solution #5**. Place the 100 mL volumetric flask below the buret, then turn the stopcock of the buret so that about **25 mL** drains into the volumetric flask. Afterwards, read the buret.	Buret Reading #5 (mL): (Include 2 decimal places!)
18. Subtract **Buret Reading #5** from **Buret Reading #4**. This number should be close to 25 mL. Record **Transfer Volume #5.** Carefully fill the volumetric flask with 0.010 M $FeCl_3$ in 0.1 M HCl solution exactly to the 100 mL mark and mix well. This is **Solution #5**. Empty the 100 mL volumetric flask into the beaker labeled "5". Rinse the volumetric flask with plenty of laboratory water.	Calculate the difference between Buret Readings #4 and #5. (Include 2 decimal places!) "Transfer Volume #5": _____ mL
19. You should now have 5 beakers labeled 1-5, each containing 100 mL of sodium salicylate at different concentrations.	

Part 2: Using an aspirin tablet to prepare a solution of unknown concentration

1. Crush one aspirin tablet in a clean porcelain mortar. Read the label of the bottle of Aspirin to find the number of GRAINS in each tablet. If Grains are not reported, record the number of grams instead.	From the Aspirin bottle, report the number of GRAINS (if not available, then report grams) each tablet should contain:
2. Transfer all the solid to a beaker (any size over 100 mL). Use several portions of 0.1 M NaOH (about 50 mL total) to rinse all the solid into the beaker. Be sure to rinse both mortar and pestle.	

3. Gravity Filtration: Prepare a folded filter paper (coffee filter paper works!) in a funnel and stand the funnel in the neck of a clean 250.0 mL volumetric flask. (The flask must be clean but does not have to be dry.) Make sure no particles of dust, paper, or other impurities get into the flask. Pour the aspirin-containing liquid from the beaker onto the filter paper in the funnel. The clear solution that comes through the filter contains your sample; the solid that is left on the filter paper will eventually be discarded. Rinse the beaker three times with 10 mL of 0.1 M NaOH (about 30 mL total) and pour these rinsings into the filter funnel also.	
4. When the sample has been filtered, remove the funnel, and dispose in the trash the filter paper and undissolved residue from the aspirin tablet.	
5. Carefully add enough laboratory water to bring the solution level exactly to the 250.0 mL line on the neck of the flask.	
6. Mix the sample thoroughly by carefully inverting the stoppered flask repeatedly for at least 2 minutes. (Hold the glass or rubber stopper tightly in place with your thumb. Make sure no liquid leaks out before the solution is completely mixed.)	
7. The solution at this point is too concentrated to be used. Prepare a dilution of it by using a volumetric pipet to transfer exactly 2.00 mL of it into a clean 100.0 mL volumetric flask (the flask may be wet as long as it is clean). Carefully add 0.010 M $FeCl_3$ in 0.1 M HCl solution to bring the solution level up to the 100.0-mL mark on the flask. Mix thoroughly.	
8. Label this 100 mL volumetric flask as “unknown”.	

Part 3: Measuring the absorption of UV light by solutions of sodium salicylate

1. The proper use and operation of the spectrophotometer can be found in Appendix: Technique I Use of Spec 20/Spec 200.
2. Adjust the spectrophotometer to measure the absorbance at 525 nm.
3. Use 0.010 M $FeCl_3$ in 0.1 M HCl solution as the reference liquid - that is, set the instrument for zero absorbance when the light path is passing through this iron(III) solution.
4. Measure the absorbance for each of your solutions of known concentrations (beakers 1-5) and for your unknown solution (the 100 mL volumetric flask labeled “unknown”). Rinse the cuvette (sample holder) thoroughly with laboratory water, then with each solution in between measurements. Record your data in the table below.

Sample	Absorbance at 525 nm
Solution #1	
Solution #2	
Solution #3	
Solution #4	
Solution #5	
Unknown	

Quick check:

- The absorbance should increase from Solution #1 (lowest) to Solution #5 (highest).
- The absorbance of the unknown should lie between the absorbances of Solution #1 and Solution #5.

Section 4: Calculations

(These may be completed after lab. However if time is available, it is recommended that you work on these calculations before leaving lab because if an error is found in your data, you may have time to obtain better data in lab.)

1. Calculate the concentration of your known solutions using the Dilution equation: $M_1V_1 = M_2V_2$.

Name of Solution	M_1 = Concentration of the solution to be transferred (the initial solution)	V_1 = Volume transferred	M_2 = Final Concentration (Calculate this number with correct SIGNIFICANT FIGURES)	V_2 = Final Volume of the solution after dilution is completed
Stock Solution	Concentration (Molarity) of the Standard Solution of sodium salicylate (from label on the standard bottle) **=**	5.00 mL		100.0 mL

Solution #1	Concentration of your Stock Solution=	Transfer Volume #1 =		100.0 mL
Solution #2	Concentration of your Stock Solution=	Transfer Volume #2 =		100.0 mL
Solution #3	Concentration of your Stock Solution=	Transfer Volume #3 =		100.0 mL
Solution #4	Concentration of your Stock Solution=	Transfer Volume #4 =		100.0 mL
Solution #5	Concentration of your Stock Solution=	Transfer Volume #5 =		100.0 mL

2. Construct the Calibration Curve

Using Microsoft Excel or another spreadsheet software package, plot the Final Concentration (y-values) vs. absorbance (x-values) for Solutions 1-5. Use Microsoft Excel to find the Line of Best Fit (this is also called Linear Regression).

Basic Instructions for Graphing with Microsoft Excel®

1. Input data into spreadsheet with x-coordinate data in the first column and y-coordinate data in the second column.
2. Select data columns with mouse.
3. Select chart wizard from either the toolbar or under the insert menu.
4. Select desired graph type. Usually xy scatter graph.
5. Select *next* button.
6. Select data range if required (usually not needed).
7. Select *next* button.
8. Input graph title and data labels.
9. Select *next* button.
10. Choose insert graph as new sheet.
11. Select data points with mouse.
12. Add trend line under chart menu or right click.
13. Select trend line type (linear).

14. Use the options tab to include the linear equation and r^2 value on the graph.

15. Make the graph look pretty using format plot area.

16. Double click axes to adjust limits.

Write the equation of the line obtained from Linear Regression (step 14 above):

_____

Ask your instructor if a plot of your data should be included with your lab report.

3. Using your equation written above, calculate the concentration of the Unknown Solution:

Sample	**Absorbance at 525 nm**	**Concentration**
Unknown		

4. From the concentration of the unknown solution, calculate the amount of aspirin in the tablet.

1. Copy the concentration of the unknown solution (from table above) that was placed into the spectrophotometer.	Concentration of the unknown solution:
2. In lab, you prepared a dilution of the unknown solution by using a volumetric pipet to transfer exactly 2.00 mL of it into a clean 100.0-mL volumetric flask. Let's calculate the concentration of that 2.00 mL solution using $M_1V_1 = M_2V_2$. M_1 = (calculate this) V_1 = 2.00 mL M_2 = Concentration of the Unknown Solution (from above) V_2 = 100.0 mL	Concentration of the unknown solution before dilution:
3. That 2.00 mL solution came from the 250.0 mL volumetric flask (this volume is equal to 0.2500 L). Calculate the number of moles of sodium salicylate in the 250.0 mL volumetric flask by multiplying 0.2500 L by the Concentration of the Unknown Solution Before Dilution	Moles of sodium salicylate:
4. For every mole of sodium salicylate in the original solution, how many moles of aspirin (acetylsalicylic acid) were there in the tablet? (See the balanced equation at the beginning of this experiment)	Moles of aspirin:
5. Calculate the number of grams from the number of moles using the molar mass of Aspirin (180.158 g/mol).	Grams of aspirin:

6. Convert grams to grains (15.43 grains = 1.000 gram).	Grains of aspirin:
7. Calculate the percent error using either grains or grams of aspirin: (Your grains of Aspirin – Bottle's grains of Aspirin) * 100 Bottle's grains of Aspirin	Percent Error:

Post-lab Questions:

1. Discuss your experimental results: do your results agree with the Aspirin manufacturer's claim regarding the grains (or grams) of Aspirin in each tablet? Discuss any of your experimental errors that may affect your conclusions.
2. Discuss how the structure of aspirin becomes more water soluble after reacting with sodium hydroxide.
3. In a different experiment, known solutions of ibuprofen were used to create a calibration curve. The line of best fit for this calibration curve was

$y = 0.000924x + 0.0000343$

where x = absorbance and y = concentration (Molarity) of ibuprofen in solution.

1. If the absorbance = 0.207, what is the concentration of ibuprofen in solution?
2. Predict the absorbance for a 5.62×10^{-4} M solution of ibuprofen.
3. Suppose an ibuprofen tablet is crushed, dissolved in sodium hydroxide, and filtered. This solution is diluted to a total volume of 5.00 L and mixed thoroughly. If this solution has an absorbance of 0.163, how many milligrams of ibuprofen were in the tablet? The molar mass of ibuprofen is 206.29 g/mol.

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1.14: Experiment_614_Synthesis of Aspirin_1_1_2

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Name	**Experiment 614: Synthesis of Aspirin**

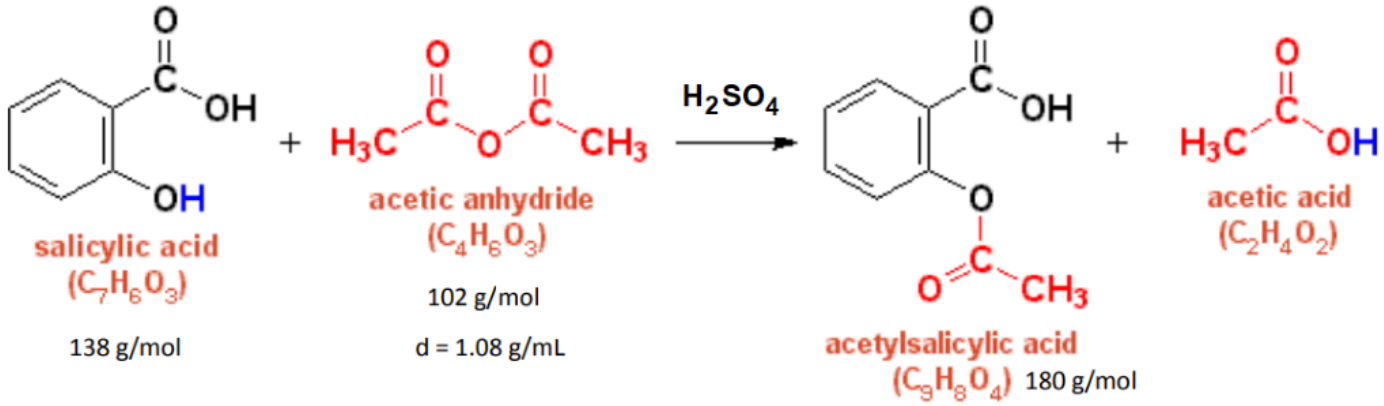
Experiment 614: Synthesis of Aspirin

Section 1: Purpose and Summary

- Conduct a chemical reaction to produce aspirin.
- Separate the aspirin from the reaction by-products using vacuum filtration.
- Analyze the aspirin and estimate its purity.

Acetylsalicylic acid, commonly known as aspirin, is the most widely used drug in the world today. Its analgesic, antipyretic, and anti-inflammatory properties make it a powerful and effective drug to relive symptoms of pain, fever, and inflammation. Salicylic acid, whose name comes from Salix, the willow family of plants, was derived from willow bark extracts. Hippocrates the ancient Greek physician, as well as Native Americans before Columbus' time, prepared willow bark teas as headache remedies and other tonics. In modern times, salicylic acid is administered in the form of aspirin which is less irritating to the stomach than salicylic acid.

To prepare aspirin, salicylic acid is reacted with an excess of acetic anhydride. A small amount of a strong acid is used as a catalyst which speeds up the reaction. In this experiment, sulfuric acid will be used as the catalyst. The excess acetic anhydride will be quenched (reacted) with the addition of water. Overall, the reaction takes place between a carboxylic acid and an acid anhydride to form an ester.



Aspirin is not very soluble in water so the aspirin product will precipitate when water is added. Some of the other compounds, acetic anhydride and acetic acid, dissolve in water, but salicylic acid is only slightly soluble in cold water. Vacuum filtration will separate the crystalline aspirin away from everything else in the reaction mixture except for any salicylic acid that did not react.

The aspirin should be analyzed for the presence of any contaminating salicylic acid. In the final part of today's lab, you will take a small amount of your aspirin and test it with iron(III) chloride ($FeCl_3$). $FeCl_3$ reacts with phenols (alcohol groups attached to aromatic rings) to produce colored complexes. Notice that salicylic acid contains the phenol functional group but aspirin does not. Therefore, the more salicylic acid that contaminates your aspirin, the darker the color will be with $FeCl_3$.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Sulfuric acid (H_2SO_4) is highly corrosive. Avoid contact with your eyes, skin, and clothing. In case of contact, rinse with plenty of water. Ask instructor to assist with the cleaning of any spills.

Acetic anhydride is a lachrymator (its vapor irritates the eyes causing tears to flow). Keep it in the fume hood.

Waste Disposal:

The reaction mixtures used in this experiment may be safely disposed of in the sink, followed by copious amount of running water.

Solids, aspirin and salicylic acid, should be disposed of in the regular trash container.

Section 3: Procedure

Part 1: Synthesis of Aspirin

1. Prepare a boiling-water bath by filling a 600-mL beaker with about 400 mL of tap water. Put the beaker on the hot plate.
2. Weigh out about 2.1 grams of salicylic acid on a piece of weighing paper. To do this, first tare a piece of weighing paper (This means to put a piece of paper on the balance and press the zero or tare button. Fig.1) Add some salicylic acid a little at a time onto the weighing paper until you have about 2.1 gram of it on the paper. It is OK to weigh a little extra mass (do not return excess salicylic acid to its container as this might contaminate the entire amount if your spatula is not perfectly clean). Record the mass of the solid. Place this solid into a 125-mL Erlenmeyer flask (other sizes may be acceptable).
3. **In the fume hood**, measure out 4 mL of acetic anhydride in a small graduated cylinder and add it to the flask. From this point on, keep your flask under the hood, because it now contains acetic anhydride (the vapors of acetic anhydride are very irritating).
4. Add about 5 drops of concentrated sulfuric acid. This will be the catalyst for the reaction.
5. Place the flask in the boiling water bath and clamp it in place. (Fig.2)
6. Heat the reaction for at least 15 minutes.
7. Put at least 60 mL of laboratory water into a 150mL beaker (or similar size). Then put this beaker in an ice-water bath. Use this cold water in steps 9, 12, 13, and 14 below.
8. After the reaction has heated for at least 15 minutes, remove it from the boiling water bath.
9. In 1 or 2 mL portions, add about 10mL cold water to the reaction, swirl the reaction between each 1-2mL portions. This water will react and destroy any remaining acetic anhydride. (Fig3.)
10. Put the flask with the reaction into an ice water bath (Fig.4). Crystals of aspirin should form. Chill for at least 10 minutes in order to obtain the maximum amount of crystalline product.



Figure 1

Figure 3

Figure 2

1. Mass of Salicylic Acid:

_____ grams

2. Volume of Acetic Anhydride:

_____ mL

3. Mass of filter paper:

_____ grams

4. Mass of crystals and filter paper:

_____ grams



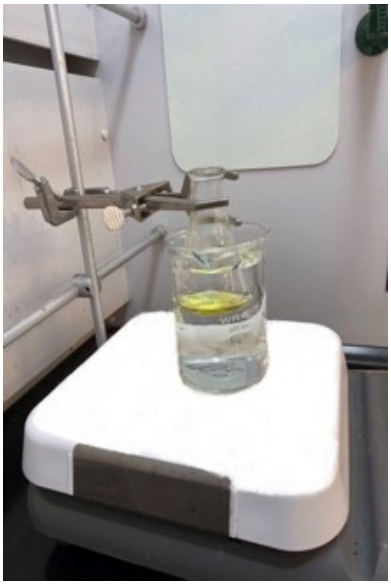


Figure 4



11. Collect the aspirin crystals by vacuum filtration.

To prepare a vacuum filtration set up:

a) You will need a Buchner funnel, a clean 250-mL vacuum filter flask, a filter adaptor (often a one-hole stopper), a pre-cut filter paper and Beaker #2. **WEIGH the dry filter paper.**

b) Place the filter paper on the Buchner funnel. The filter paper should fit snugly and cover all the small holes in the funnel.

c) Using the filter adaptor, place the Buchner funnel on top of a vacuum filter flask. You might need to use a clamp and a ring-stand to keep the setup intact and upright.

d) Connect a vacuum tubing hose to the vacuum filter flask and to the vacuum supply.



Figure 5

e) Wet the filter paper with a little laboratory water. Turn the vacuum on and confirm that there are no leaks.(Fig.5)

12. Add about 25 mL cold water to the reaction and mix well.
13. Filter the crystals. Use a few milliliters of cold water to transfer as much solid as possible.
14. Disconnect the vacuum temporarily and add 15 mL of cold water to the crystals in order to rinse them. After the crystals have soaked in the cold water for a few seconds, reconnect the vacuum to filter once more. Leave the vacuum on for several minutes to air dry the crystals.
15. With the vacuum on, stir the crystals carefully to help dry them. Do this for about 15 minutes.
16. Turn off the vacuum and weigh just the filter paper with the crystals. If you use a watch glass to carry the filter paper and crystals to the balance, be sure to tare the weight of the watch glass (in other words, don't include the weight of the watch glass with your filter paper and crystals.)
17. Clean up: Be sure to keep the aspirin for Parts 2 & 3. Throw away the filter paper in the trash. Dispose of the filtrate (the aqueous solution in the vacuum filtration flask) down the drain with plenty of water. In the fume hood, rinse with water all pipets and glassware in contact with acetic anhydride and pour the rinse water down the drain in the fume hood.

Part 2: Analysis of Aspirin (How pure is it?)

1. Obtain 3 test tubes and about 1 mL of ethanol (CH_3CH_2OH) to all 3 test tubes. Also add 1-2 drops of 1% $FeCl_3$ solution to each test tube. Be sure all 3 test tubes have the same amounts of reagents.	
2. Label the first test tube "Aspirin" and add to this test tube a few of your aspirin crystals that you made today. Stir well.	**Describe the Color:**
3. Label the second test tube "Salicylic Acid" and add a few crystals of Salicylic Acid to this second test tube. Stir well. This is your **Positive Control**. The positive control shows you what to look for when a test is positive: in today's analysis, the positive control shows you a dark color because it contains unreacted salicylic acid.	**Describe the Color:**

4. The third test tube is your **Negative Control**. Do not add any crystals to this test tube! The negative control shows you what to look for when a test is negative: in today's analysis, the negative control shows you a color when no contaminating salicylic acid is present.	**Describe the Color:**
Compare the colors among the 3 test tubes and describe their similarities and differences.	**Describe the similarities and differences in color:** **(You can use the empty space to the left also).**

Part 3: Analysis of Aspirin (How stable is it?)

1. Obtain a dry test tube and add about a pea-sized amount of your aspirin crystals (borrow some relatively pure crystals from a classmate if your aspirin was highly contaminated with salicylic acid). 2. Tap the crystals to the bottom of the test tube. Use a test tube clamp to hold the test tube. 3. Ignite a Bunsen burner. 4. Pointing the open end of the test tube away from you, GENTLY heat the bottom of the test tube until the crystals melt. DON"T burn the aspirin.(Fig.5) 5. Remove the test tube from the flame and carefully WAFT any odors from the open end of the test tube towards you. Do you smell anything? (To waft the odors means to "fan" the odors towards you, mixing in plenty of air to dilute the fumes.) If you don't smell anything, try "pouring" the vapors out of the test tube by holding the test tube horizontally. 6. Any odor detected may indicate that aspirin has decomposed with excessive heat. Aspirin may also decompose slowly upon long-term storage in a humid environment. 7. After the test tube cools, wash the test tube in the sink with soap and water.	**Describe the Odor:**

Section 4: Calculations

1. Mass of salicylic acid (g)	
2. Calculate the number of moles of salicylic acid (mol) (Molar mass = 138 g/mol)	
3. Volume of acetic anhydride (mL)	

4. Calculate the number of grams of acetic anhydride (g) (Density = 1.08 g/mL)	
5. Calculate the number of moles of acetic anhydride (mol) (Molar mass = 102 g/mol)	
6. Identify the Limiting Reagent (Name of compound)	
7. Theoretical Yield of Aspirin (g) (Molar mass = 180 g/mol)	
8. Mass of filter paper and aspirin (g)	
9. Mass of filter paper (g)	
10. Calculate the mass of aspirin (g)	
11. Percent Yield of Aspirin (%)	

Post Lab Questions:

1. Aspirin is slightly soluble in water: the solubility of aspirin in water is 0.33 grams per 100 mL water at room temperature. In today's experiment, you rinsed your aspirin with at least 50 mL of water. If the water was at room temperature, how many grams of your aspirin would have dissolved by rinsing it today?
2. Suggest a reason why you were instructed to rinse the aspirin even though it is known that this will cause some of the aspirin to dissolve and be lost.
3. Suppose that another student performed today's experiment, but they forgot to air dry the aspirin during vacuum filtration. If they weigh the aspirin when it is still damp, how will this affect the percent yield?
4. Suggest a scientific reason why we did not dry the aspirin in an oven today.
5. A desiccant, or drying agent, is often added to a container with medications to prolong the shelf life. Cotton is sometimes used as a desiccant which is often found inside a package of aspirin. Moisture in the air (humidity) can cause aspirin to slowly decompose through a reaction called hydrolysis. The cotton absorbs moisture and delays hydrolysis. Suggest an easy and quick way to determine if your aspirin at home has begun to hydrolyze.
6. Suppose two groups of students performed today's experiment and obtained different results. One group obtained aspirin in 88% yield which turned dark in the $FeCl_3$ test. The other group obtained aspirin in 65% yield which produced no color change in the $FeCl_3$ test. Explain which group was more successful in lab.

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1.15: Experiment_615_Titration of Vinegar_1_2_3

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 615: Titration of Vinegar**

Experiment 615: Titration of Vinegar

Section 1: Purpose and Summary of Experiment

In this experiment, students will determine the precise concentration of a weak acid solution that has an unknown molarity. Students will do this by performing a *titration*. A titration is an experimental technique for determining the concentration of a solution by using a chemical reaction with another solution.

Titration is a versatile and relatively inexpensive investigative procedure to determine a concentration of a substance. Accuracy and precision better than 1% are commonly achieved. It is used daily in many industries, sometimes with automation, to monitor manufacturing as part of quality control. Dozens of variations of titrations exist.

To do a titration, three things must be known:

- A balanced chemical equation that describes the chemical reaction being used
- A way of indicating when the reaction has been completed
- A standard, known amount of one reactant

To perform a titration, a carefully measured amount of one reactant is added to a reaction flask. Usually an Erlenmeyer flask is used as a reaction flask. An *indicator* is added that will signal the endpoint of the titration by a visible color change when the reaction is complete. The other reactant, called a *titrant* is added slowly to the flask using a *buret* just until one drop of the titrant causes the indicator to change color. When the indicator changes color, the reaction is complete and the volume of the titrant added is measured. This is called the *equivalence point*, because it occurs when the reaction is complete. If you know the volumes of both solutions used and the concentration of one of the solutions, you can calculate the concentration of the other solution using stoichiometry.

NOTE: Equivalence point (when the reaction is complete) and endpoint (when the indicator has changed color in a titration) have slightly different meanings. Indicators are selected so that the endpoint occurs close to the equivalence point. The terms are often used interchangeably.

This is a key point: *it is important to add only just enough titrant to change the indicator*. The indicator changes at the endpoint. Once the indicator has changed, it should not change back. Addition of additional titrant will introduce error in the determination.

Vinegar is a complex mixture that contains acetic acid as its acidic component. Using a vinegar sample, you will determine the precise concentration of an acetic acid (CH_3COOH) solution by titration. Because acids will react with bases, you will use a solution of sodium hydroxide (NaOH). The concentration of the NaOH standard solution has been determined by a procedure known as s*tandardization* and is known very precisely, usually to four significant figures.

Students will use a standardized NaOH solution to titrate a sample of vinegar. The reaction is as follows:

$CH_3COOH(aq) + NaOH(aq) \rightarrow H_2O(l) + NaCH_3COO(aq)$	**Reaction A**

Acetic acid		Sodium hydroxide		Water		Sodium acetate	

Use the titration data to calculate the molarity of acetic acid in the vinegar.

Try a sample calculation. Refer to Section 4 for sample calculations:

A 5.00 ml sample of vinegar was titrated with 0.08751 M NaOH. The sample requires 22.31 mL of NaOH to reach the endpoint. Calculate the molarity of the acetic acid (CH_3COOH) solution in the vinegar sample. The density of the vinegar solution has been determined to be 1.0052 g/ml.

If the vinegar is being manufactured, and is supposed to have a molar concentration between 0.45 and 0.60, can this batch be sold?

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Wear your safety goggles.

If any acid or base solution splashes on you, rinse it off immediately.

The indicator used in this experiment is called phenolphthalein. Phenolphthalein has laxative properties and was once used medically for that purpose. Wash your hands and do not eat in the laboratory.

Never use your mouth to provide suction to a pipet. Always use a suction device like a rubber bulb.

Waste Disposal:

Waste from this experiment may be safely discarded down the drain using plenty of running water.

Section 3: Procedure

Note: Transfer pipets (sometimes called volumetric pipets) and burets are usually made of glass and are precision instruments. Please use gently, because they can and do break. If you are trying to put force on glass, stop and ask your instructor for assistance.

In this lab, as in any precise titration, you must be very careful not to alter the concentration of the solutions in any way before their volumes have been measured. Glassware used to measure solutions must be both absolutely clean and dry or rinsed with 3 small portions of the solution to be used in it (being sure to wet the entire inner surface of the glassware each time). If you accidentally use a piece of glassware that is already wet with laboratory water, the small amount of water on the inside of the glassware will dilute the solution slightly and alter its concentration. This may introduce error and you might have to start the process over. This is true for precision pipets and burets.

Refer to the balanced chemical equation of this system. Notice that water is not a reactant in **Reaction A**, although it is produced as a product. The addition of extra water does not change the number of reactant molecules in the reaction flask once the volumes of the solutions have been precisely measured and dispensed. For this titration, then it is perfectly fine to use laboratory water as necessary for washing the sides of the reaction flask.

Part 1: Cleaning a Volumetric or Transfer Pipet (Refer to Technique E)

1. Pour approximately 50 ml of a soap solution in a 100-ml beaker.

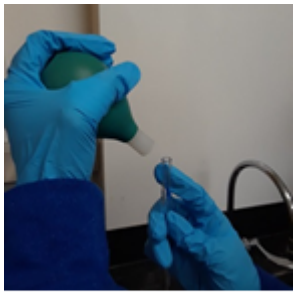
2. Using a rubber suction bulb, lower the pipet tip beneath the surface of the soap solution and draw enough soap solution into the pipet to fill it about one-third full. Remember to keep the pipet tip below the surface of the solution. *Be careful as you get to the top of the middle section of the transfer pipet--the solution uptake will get faster!!*
3. Release the pressure on the bulb slowly to avoid drawing solution into the bulb. Quickly and carefully disconnect the bulb from the pipet. *Immediately* place your index finger over the open top of the pipet in order to prevent the solution from draining out. Hint: If you use your index finger, not your thumb, you will have more control over the level of the liquid in the transfer pipet.
4. Hold the pipet almost horizontally. Carefully rotate the pipet so that the soap solution contacts the entire inner surface of the pipet. Remember to remove your index finger from the top of the pipet to allow the soap solution to fill the upper stem of the pipet.
5. Drain the soap solution through the pipet tip into the sink.
6. Rinse the pipet several times with laboratory water using the same procedure as in steps 2-5.
7. Fill a beaker with laboratory water. Fill the whole pipet one time as a final rinse of the interior surface of the pipet.
8. Repeat for any and all pipets you are using for this experiment.

Part 2: Preparing the Vinegar Sample for Titration

1. Obtain about 100 ml of a vinegar sample in a clean, dry 250-ml beaker.
2. Pour approximately 40 ml of the vinegar sample into a clean, dry 150 ml beaker. Rinse your clean pipets from Part 1 two times with about 5 ml of your vinegar sample each time using techniques similar to Part 1 steps 2-5. Your vinegar rinse waste can be discarded down the drain with running water.

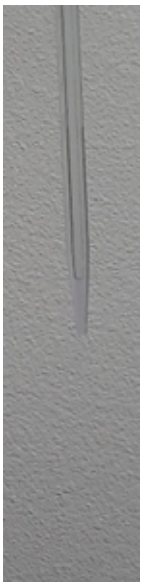
3. Pipet 25.00 ml of your vinegar sample into a clean 250-ml Erlenmeyer flask. Record the volume of vinegar transferred on Part 5: Data Table line 1. If you use a transfer pipet with a different volume (10.00 ml, 15.00 ml and 20.00 ml are common), record the actual volume used. An example of the proper use of a transfer pipet is here:

Use a rubber bulb for suction. There are different styles of rubber bulbs. The style shown does not stay on the pipet. Use it for suction and then quickly use your finger to control the height of the column of solution in the transfer pipet. Pipet the fluid above the calibration mark. The fluid in the photos has been dyed purple to make it easier to see:





Position your eye at the same level as the calibration mark. If the calibration mark looks like an oval, your eye is not at the correct level. Let fluid fall until the meniscus just touches the paint line of the calibration mark:

 Correct	 Not Correct (Paint line is an oval and meniscus is not level)	 Leave the last drop

4. Add about 30-40 ml of laboratory water from a clean 50-ml graduated cylinder to the vinegar sample in the Erlenmeyer flask. This is just to add volume to this solution.

5. Add 1 drop of phenolphthalein indicator solution to the Erlenmeyer reaction flask. Gently swirl the flask and its contents to thoroughly mix the solution.

Part 3: Cleaning and Filling the Buret (Refer to Technique G)

1. Place the buret on a support stand using a buret clamp. A photo of the typical setup looks like this:



2. Close the stopcock and place a *buret funnel* (a small funnel that fits loosely in the top of the buret) at the top of the buret. Add 10 ml of laboratory water to the buret through the funnel. Make sure the buret does not leak. Notify your laboratory instructor if the buret leaks.

3. Remove the buret from the support stand. Hold the buret almost horizontally. Carefully rotate the buret so that the water contacts the entire surface.

4. Drain the water through the tip into the sink.

5. Repeat steps 1-3 twice using 10 ml of laboratory water each time.

6. Obtain about 100 ml of the NaOH standardized solution in a clean, dry 250-ml Erlenmeyer flask. Record the molarity of the NaOH solution in Part 5: Data Table line 2. It usually takes some time to standardize a solution, so take what you need, but use the solution conservatively.

7. Repeat steps 1-3 three times using 5 ml of the NaOH standard solution each time.

8. Finally, put a little of the NaOH solution in the buret and flush out any air from between the stopcock and the buret tip.

9. Clamp the empty buret to the support stand.

10. Fill the buret with NaOH solution to the 0.00 ml line at the top of the buret. The meniscus should be blocked by the paint-line of the 0.00 ml mark. If you miss the 0.00 ml mark, it is not a problem. Just read the meniscus to the nearest 0.01 ml and record this initial reading on Part 5: Data Table line 3. You can subtract this initial volume out later.

Part 4: Titrating Vinegar

1. Place the 250-ml Erlenmeyer flask containing the vinegar solution under the buret tip. Lower the buret tip so the tip extends about 1 cm into the mouth of the flask. Keeping the buret tip below the level of the flask reduces the likelihood of any droplets splashing out of the flask.
2. Open the stopcock and release 1-2 ml volumes of the NaOH solution from the buret to the vinegar solution while gently swirling the flask. You should see a pink coloration in the solution at the point where the NaOH solution contacts the surface of the vinegar solution. That is the color of the indicator. As you approach the endpoint of the titration, the pink will begin to momentarily flash through the entire solution. When you see this happening, slow down and begin adding the NaOH solution one drop at a time. You can even add 1/2 drop by dispensing a little of the NaOH on the pipet tip and washing it in using laboratory water.
3. Stop the titration when pink persists throughout the entire solution for 30 seconds after you have thoroughly swirled the flask. You have now reached the endpoint. Record the reading on the buret to the nearest 0.01 ml on Part 5: Data Table line 4.
4. Refill the buret with your NaOH solution to the 0-ml line at the top of the buret. Do a second determination by preparing a new vinegar sample as in Part 2 Steps 3-5. Then repeat Part 4 steps 1-3 to record a second set of data in column #2.
5. When finished, discard the remaining NaOH solution in the buret into the sink with running water.
6. Rinse the empty buret twice with 10 ml of laboratoy water each time.
7. Discard all remaining solutions into the sink with running water.

Part 5: Data Table

	1
1. Volume of vinegar sample (ml)	
2. Molarity of NaOH standard solution (M)	

3. Initial buret reading (ml)	
4. Final buret reading (ml)	

Section 4: Calculations

Part 1: Determining the molarity of the vinegar sample

1. Volume of NaOH used (ml)	
2. # of moles of NaOH required for titration (mol)	
3. # of moles of CH_3COOH in vinegar sample (mol)	
4. Molarity (mol/L) of CH_3COOH in vinegar sample (M)	
5. Mean (the average of multiple values) molarity of CH_3COOH in vinegar sample (M)	

Calculating # of moles from molarity and volume

#moles of NaOH required for titration = $\left(\begin{array}{c}\text{volume of NaOH}\\ \text{solution used, ml}\end{array}\right)\left(\frac{1L}{1000\text{ml}}\right)\left(\begin{array}{c}\text{concentration of}\\ \text{NaOH solution, M}\end{array}\right)$

 # moles of CH_3COOH in vinegar sample = $\left(\begin{array}{c}\text{number of moles of}\\ \text{NaOH required, mol}\end{array}\right)\left(\frac{1\text{ mol }CH_3\text{ COOH}}{1\text{ mol }NaOH}\right)$

Molarity of CH_3COOH in vinegar sample = $\left(\frac{\text{number of moles of CH}_3\text{COOH, mol}}{\text{volume of vinegar sample, }L}\right)$

NOTE: The fraction 1 mol CH_3COOH / 1 mol NaOH is derived from the ratios in the balanced chemical equation for this reaction.

Post Lab Questions:

1. Briefly explain why adding laboratory water to the vinegar sample in the reaction flask prior to beginning the titration does not affect the results of your determination of the molarity of CH_3COOH in your vinegar sample.
2. A 25.00 mL sample of a sulfuric acid solution (H_2SO_4) is titrated with sodium hydroxide (NaOH). If it requires 35.88 mL of 0.1127 M NaOH to reach the endpoint, what is the molarity of the sulfuric acid solution? (All of the hydrogens of the sulfuric acid react with the sodium hydroxide.) Hint: Write a balanced chemical equation of this reaction first.
3. The vinegar in this titration was distilled vinegar and is clear and colorless. What modifications would you need to do to this procedure in order to test solutions such as cider vinegar or pickling liquor, both of which have significant color?

Notes:

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1.16: Experiment_616_Shifting Equilibrium_1_1_3

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 616: Shifting Equilibrium**

Experiment 616: Shifting Equilibrium

Section 1: Purpose and Summary

Determine the effect of different types of stresses to a chemical system at equilibrium.

In this experiment, students will determine the effect of adding or removing a reactant or product, or heating/cooling to a chemical system at equilibrium and apply Le Chatelier's principle to explain the shift in the position of equilibrium.

The following chemical systems will be studied:

1. saturated NaCl solution: $NaCl(s) \leftrightarrow Na^{+}(aq) + Cl^{-}(aq)$
2. acidified chromate solution: $2\ CrO_4^{2-}(aq) + 2\ H^{+}(aq) \leftrightarrow Cr_2O_7^{2-}(aq) + H_2O(l)$

yellow orange

3. aqueous ammonia solution: $NH_3(aq) + H_2O(l) \leftrightarrow NH^{4+}(aq) + OH^{-}(aq)$

with phenolphthalein clear pink

4. cobalt(II) chloride solution: $[Co(H_2O)_6]^{2+}(aq) + 4\ Cl^{-}(aq) \leftrightarrow [CoCl_4]^{2-}(aq) + 6\ H_2O(l)$

pink blue

5. iron(III)thiocyanate solution: $Fe^{3+}(aq) + SCN^{-}(aq) \leftrightarrow [Fe(SCN)]^{2+}(aq)$

lt. yellow clear deep red

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Waste Disposal:

While you are doing the experiment, pour your liquid waste into a beaker. When you are finished with the experiment, pour the contents of the waste beaker (liquid waste only) into the inorganic waste container in the fume hood.

Section 3: Procedure

To determine whether a reaction occurs or not, observe any color changes or precipitates. Note that the volumes of the solutions indicated are approximate.

Part 1: Saturated sodium chloride solution

1. Transfer about 3 mL of saturated NaCl solution into a small test tube.
2. Using a dropper, carefully add concentrated (12 M) HCl drop by drop to the saturated NaCl solution until a distinct change is observed. Record your observations.

Equilibrium system chemical equation:

Observations after addition of HCl:

In which direction (left or right) did the addition of HCl cause the equilibrium to shift?

Which ion caused the shift? Explain.

Part 2: Acidified chromate solution

1. Transfer about 3 mL of aqueous 0.1 M potassium chromate (K_2CrO_4) solution into a small test tube. Note the color of the solution.
2. Add an equal amount (3 mL) of 6 M nitric acid (HNO_3). Record your observations.
3. Then, add 10% aqueous NaOH solution drop by drop until a color change is observed. Record your observations.

Equilibrium system chemical equation:

Observations after addition of HNO_3:

In which direction (left or right) did the addition of HNO_3 cause the equilibrium to shift?

Which ion caused the shift? Explain.

Observations after addition of NaOH:

In which direction (left or right) did the addition of NaOH cause the equilibrium to shift? **Which ion caused the shift? Explain.**

Part 3: Aqueous ammonia solution

For Stockroom notes only: Preparation of ammonia stock solution: Transfer 4 drops of concentrated (15 M) NH_3 solution and 3 drops of phenolphthalein indicator into a 150-mL beaker. Add 100 mL laboratory water and mix thoroughly using a stirring rod.

NOTE: Aqueous ammonia solution is sometimes written as NH_3 or as NH_4OH.

1. Transfer about 3 mL of the prepared ammonia stock solution into a small test tube.
2. Add about 1 cm^3 ammonium chloride (NH_4Cl) powder into the solution. Record your observations.

Equilibrium system chemical equation:
Observations after addition of NH_4Cl:
In which direction (left or right) did the addition of NH_4Cl cause the equilibrium to shift? **Which ion caused the shift? Explain.**

Part 4: Aqueous cobalt(II) chloride solution

1. Label three small test tubes (#1, #2, and #3).
2. Transfer about 3 mL of aqueous cobalt(II) chloride solution into each numbered test tube.
3. The solution in test tube #1 is the control. All observations to be made on test tubes 2 and 3 will be compared with test tube #1.
4. To test tube #2, carefully add concentrated (12 M) HCl solution drop by drop until a distinct color change occurs. Record your observations.
5. To test tube #3, add about 1 cm^3 ammonium chloride (NH_4Cl) powder. Heat the mixture directly over a Bunsen burner, using a moderate flame. In a slant position, move the test tube back and forth for about 30 seconds, or until a distinct color change occurs. Record your observations. Let the solution cool to room temperature and observe. Record any observed changes.

Equilibrium system chemical equation:
Observations after addition of HCl:
In which direction (left or right) did the addition of HCl cause the equilibrium to shift? **Which ion caused the shift? Explain.**

Observations after heating and cooling:
In which direction (left or right) did heating cause the equilibrium to shift? **In which direction (left or right) did cooling cause the equilibrium to shift?** **Based on these observations, is this reaction (as written) endothermic or exothermic? Explain.**

Part 5: Aqueous iron(III) thiocyanate solution

For Stockroom notes only: Preparation of iron(III) thiocyanate stock solution: Combine 1 mL of 0.1 M iron(III) chloride ($FeCl_3$) solution and 1 mL of 0.1 M potassium thiocyanate (KSCN) solution in a 150-mL beaker. Add 100 mL of laboratory water and mix thoroughly using a stirring rod.

1. Label four small test tubes (#1, #2, #3, and #4).
2. Transfer about 3 mL of stock iron(III) thiocyanate solution into each numbered test tube.
3. The solution in test tube #1 is the control. All observations to be made on test tubes 2 to 4 will be compared with test tube #1.
4. To test tube #2, add 1 mL of 0.1 M iron(III) chloride ($FeCl_3$) solution. Record your observations.
5. To test tube #3, add 1 mL of 0.1 M potassium thiocyanate (KSCN) solution. Record your observations.
6. To test tube #4, add 0.1 M silver nitrate ($AgNO_3$) solution drop by drop until a distinct color change occurs. Record your observations. Note that thiocyanate ion reacts with silver ion to form a precipitate: $SCN^-(aq) + Ag^+(aq) \rightarrow AgSCN(s)$

Equilibrium system chemical equation:

Observations after addition of $FeCl_3$:
In which direction (left or right) did the addition of $FeCl_3$ cause the equilibrium to shift? **Which ion caused the shift? Explain.**

Observations after addition of KSCN:
In which direction (left or right) did the addition of KSCN cause the equilibrium to shift? **Which ion caused the shift? Explain.**

Observations after addition of $AgNO_3$:
In which direction (left or right) did the addition of $AgNO_3$ cause the equilibrium to shift? **Which ion caused the shift? Explain.**

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1.17: Experiment_617_pH_Acids_and_Bases_1_1_3

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 617: pH, Acids, and Bases**

Experiment 617: pH, Acids, and Bases

Section 1: Purpose and Summary

- Understand relationship between pH and H^+ ion concentration
- Understand pH differences of acids and bases
- Determine the pH of common solutions
- Show how to use a pH indicator

A pH value is a number, usually between 0 and 14, that provides information about how acidic or basic a solution is. The “pH” is always written with a lowercase “p” and an uppercase “H”, which stands for “power of hydrogen.” pH values are related to hydrogen ion (H^+) concentrations.

The mathematical relationship between pH and the molar concentration of H^+ is described by the equation:

pH = - log [H^+]

where log is the common log (the log of base 10) and [H^+] is the molar concentration (i.e., moles of H^+ per liter).

NOTE: It may be a good time to review logarithmic relationships. The fundamental ones you need to know are:

$\log_b(x) = y$ *if* $b^y = x$ and $-\log_b(x) = \frac{1}{\log_b(x)}$

Because the negative of the log is being used, there is an inverse relationship between pH and [H^+] ion concentration. As the [H^+] concentration decreases, the pH value increases, and vice versa. If you are using common logs, the base of the logarithm is not usually shown.

When the pH value is a whole number (for example, a solution of pH 7), the whole number is equal to the negative exponent of the [H^+] ion concentration.

Example: 10^{-7} = [H+] = 10^{-7} moles of H+ per liter of solution.

pH = -log (10^{-7})

pH = - (-7)

pH = 7

The pH values of everyday chemicals typically range from pH 0 to pH 14. Values between 0 and 7 indicate an acidic solution. Values between 7 and 14 indicate a basic solution. A pH of exactly 7 indicates that a solution is neutral; it is neither acidic nor basic.

Pure water is usually pH 7. The pH scale is shown below.

0 1 2 3 4 5 6 7 8 9 10 11 12 13 14

acidic neutral basic

(alkaline)

The lower the pH value, the more acidic the solution; the higher the pH value, the more basic the solution. Basic solutions are also called alkaline solutions. The pH scale can extend beyond 0 and 14, but these values are found in extreme cases. Strong, concentrated laboratory acids can have pH values less than 0 (negative pH values) and strong, concentrated laboratory bases can have pH values greater than 14. Solutions at extreme pH values are considerably more reactive in general and must be used with additional care.

The concept of pH is widely used in all areas of science including agriculture, biology, engineering and medicine. Many commercial products use pH as an advertisement tool, such as shampoo and water. Recently, food and drinks of certain pH has been touted as more healthful.

A pH indicator is a substance that, when a small amount of it is added to a solution of unknown pH, will have a color that depends upon the pH. This is a way to determine pH of a solution visually. pH indicators are a good way to easily and quickly show the approximate pH by color when compared to a standard. An everyday example where a pH indicator is used is for testing a water sample from a swimming pool. In the following table, you can see a variety of indicators with the approximate pH range over which each changes color.

Indicator	*pH range*	*Color change*
Methyl violet	0.0 to 1.6	Yellow to violet
Cresol Red (1)	0.2 to 1.8	Red to yellow
Thymol blue (1)	1.2 to 2.8	Red to yellow
Cresol purple (1)	1.2 to 2.8	Red to yellow
Methyl orange	3.1 to 4.4	Red to yellow
Methyl red	4.8 to 6.0	Red to yellow
Bromocresol green	3.8 to 5.4	Yellow to blue
Litmus	5.0 to 8.0	Red to blue
Bromothymol blue	6.0 to 7.6	Yellow to blue
Phenol red	6.4 to 8.0	Yellow to red

Cresol red (2)	7.2 to 8.8	Yellow to red
Cresol purple (2)	7.6 to 9.2	Yellow to purple
Phenolphthalein	8.0 to 9.6	Colorless to pink
Thymolphthalein	8.3 to 10.5	Colorless to blue
Thymol blue (2)	8.0 to 9.6	Yellow to blue
Alizarin yellow	10.1 to 12.0	Yellow to orange-red

Three indicators in the table above (cresol red, thymol blue and cresol purple) change colors in two different pH ranges. Indicators are usually identified by the common name.

1. Which indicator(s) in the table above would be useful to estimate the pH range of a strongly acidic solution?

Which indicator(s) in the table above would be useful to estimate the pH range of a strongly basic solution?

Which indicator(s) in the table above would be useful to estimate the pH range of a more neutral solution?

2. Thymol blue changes colors in 2 different pH ranges. If the solution you test with the thymol blue indicator turns yellow, would you be able to conclude whether that solution is acidic or basic? Why or why not?

3. You are given an unlabeled test tube and you need to find an approximate pH range of the solution in the tube. You are given the following data:

Indicator	*Indicator color in solution*
Cresol Red	Yellow
Methyl orange	Yellow
Phenolphthalein	Pink

What is the pH range you can identify for the solution in the unknown test tube? What evidence leads you to this conclusion?

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Wear your safety goggles.

If any acid or base solution splashes on you, rinse it off immediately and notify your laboratory instructor. If any acid or base solution spills on a work surface, immediately notify your instructor.

Waste Disposal:

Waste from this experiment may be safely discarded down the drain using plenty of running water.

Section 3: Procedure

It is important to keep solutions from cross contaminating each other during this experiment. The best technique is to use a glass stir rod to get a drop of solution. You can rinse off the stir rod with laboratory water and dry it between tests.

Part 1: Estimating the pH of an Aqueous Solution of Nitric Acid (HNO_3)

1. Label each of 3 test tubes with a number #1, #2, or #3.
2. Obtain 9 ml of a 0.01M nitric acid (HNO_3) solution in a clean, dry 10-ml graduated cylinder. Pour 3 ml of the solution into each of your numbered test tubes. Use a test tube rack or stand to hold your test tubes upright.
3. Rinse the graduated cylinder twice with 5 ml of tap water each time followed by 5 ml of laboratory water.
4. Place one strip of blue litmus paper and one strip of red litmus paper on a clean, dry watch glass. Keep the two pieces of litmus paper well separated on the watch glass.
5. Dip the tip of a clean glass stirring rod into the (HNO_3) solution in one of your test tubes. Transfer that drop to the strip of blue litmus paper by touching the rod with the drop to one end of the blue litmus paper. Note the color and record your observations on Data Sheet line 1.
6. Repeat step 5 but transfer the drop from the stirring rod to the strip of red litmus paper. Note the color and record your observations on Data Sheet line 2.
7. Discard the used litmus paper in the garbage and rinse the stirring rod with laboratory water.
8. Add 3 drops of thymol blue indicator solution to the solution in test tube #1. Note the color, if any, and record your observations on Data Sheet line 3.
9. Add 3 drops of methyl orange indicator solution to the solution in test tube #2. Note the color, if any, and record your observations on Data Sheet line 4.
10. Add 3 drops of phenolphthalein indicator solution to the solution in test tube #3. Note the color, if any, and record your observations on Data Sheet line 5.
11. Discard the contents of your test tubes down the drain with plenty of running water. Rinse the test tubes twice with 5 ml of tap water followed by 5 ml of laboratory water. Dry the test tubes (and keep the labels or re-label as needed) to use in Part 2.

Part 2: Estimating the pH of Solutions of Various Substances

Note: Your laboratory instructor will assign solutions from the following list for you to study.

1. Soda
2. Shampoo
3. Ammonia cleaner
4. Bleach
5. Laundry detergent
6. Lemon juice
7. Vinegar
8. Bottled water

1. Obtain a 9 ml portion of the assigned solution in a clean, dry 10-ml graduated cylinder. Pour 3 ml of the solution into each of your dry, numbered test tubes.
2. Test the solution in one of the test tubes with blue litmus paper and red litmus paper following the same procedure as in Part 1 lines 5 and 6. Record your observations on Data sheet column 6-7 next to the formula of your assigned solution.
3. Add 3 drops of thymol blue indicator solution to the solution in test tube #1. Record your observations on Data sheet column 8 next to the formula of your assigned solution.
4. Add 3 drops of methyl orange indicator solution to the solution in test tube #2. Record your observations on Data sheet column 9 next to the formula of your assigned solution.
5. Add 3 drops of phenolphthalein indicator solution to the solution in test tube #3. Record your observations on Data sheet column 10 next to the formula of your assigned solution.
6. Discard your solutions and clean and dry your test tubes as you did in Part 1 line 11.
7. Repeat Part 2 steps 1-6 for any remaining assigned solutions and record your data in columns 9-13 next to the solution you tested.

Part 3: Data Tables

pH of Nitric Acid (HNO_3)	*Observations*
1. Blue litmus	

2. Red litmus	
3. Thymol blue	
4. Methyl orange	
5. Phenolphthalein	

pH of various substances	*Observations*		
0.1M solutions	*Blue litmus (6)*	*Red litmus (7)*	*Thymol bl*
1. Soda			
2. Shampoo			
3. Ammonia cleaner			
4. Bleach			
5. Laundry detergent			
6. Lemon juice			
7. Vinegar			
8. Bottled water			

Is an aqueous solution of HNO_3 acidic or basic? What evidence supports your conclusion?

List each of the tested substances that are acidic. What evidence supports your selections?

List each of the tested substances that are basic. What evidence supports your selections?

Arrange the tested substances in order from most acidic to most basic.

Post Lab Question:

The labels fell off of three test tubes, each containing a colorless solution. A student knew that one of the test tubes contained a

0.1M HCl solution (acid), another contained a 0.1M NaOH solution (base), and the third contained a 0.1% phenolphthalein solution (indicator). To determine the identity of each solution, the student temporarily labeled the test tubes “A”, “B”, and “C”. The student mixed the solutions in various combinations, recording the observations in the table below:

Observations after mixing three unknown solutions

Mixture observations

1. 1 drop of “A” + 1 drop of “B” red solution
2. Solution from (1) + 1 drop of “C” solution became colorless
3. Solution from (2) + 1 drop of “B” solution turned red

Determine which original label (with chemical identity) should be attached to each of the test tubes. Justify your answer briefly.

Notes:

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1.18: Experiment_619_Heat of Solution_1_1_3

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 619: Heat of Solution**

Experiment 619: Heat of Solution

Section 1: Purpose and Summary

- Determine the heat of solution of two ionic compounds: ammonium chloride and calcium chloride.
- Determine which dissolution process is endothermic and which one is exothermic.

Physical processes, like dissolution, involve heat changes. Some release heat, while others absorb heat. In this experiment, students will determine the amount of heat involved when ammonium chloride (or calcium chloride) is dissolved in water. The calculation is based on the calorimetry equation:

Heat given off or absorbed = (mass)(specific heat)(change in temperature)

$q = m \times c \times \Delta T$

The heat of solution (ΔH_{soln}) is the energy involved in dissolving a specific amount of solute in a given solvent. A process that gives off heat is called exothermic ($-\Delta H_{soln}$), and a process that absorbs heat is called endothermic ($+\Delta H_{soln}$). Based on observations and calculations, students will classify which dissolution process is endothermic and which is exothermic.

$\Delta H = q$ / n where n = mole of limiting reagent

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Waste Disposal:

The reaction mixtures used in this experiment may be safely disposed of in the sink, followed by copious amount of running water.

Section 3: Procedure

Part 1: Dissolution of ammonium chloride

1. Obtain two Styrofoam cups. Construct a simple calorimeter by nesting one cup inside the other. Weigh the cups. Record exact mass.	1. Mass of cups: _____ grams

2. Measure 100 mL laboratory water using a graduated cylinder, and transfer into the Styrofoam cup. Weigh again. Record exact mass.	2. Mass of water + cups: _____ grams
3. Cover cup with a lid with two holes – one for inserting a thermometer and the other for a stirring rod. Record the temperature of the water to ±0.1°C. This is the initial temperature (T_i).	3. Initial temperature, T_i (°C):
4. Using the tare function, weigh about 20 grams of solid ammonium chloride (NH_4Cl) on a weighing paper/ boat. Record the exact mass.	4. Mass of NH_4Cl: _____ grams
5. Transfer the ammonium chloride into the Styrofoam cup. Put the lid back, quickly insert the thermometer and stirring rod, and stir the mixture vigorously. Be careful not to hit the thermometer while stirring. Immediately record the temperature of the mixture to ±0.1°C. Continue stirring. Record the temperature of the water at 30 second intervals for 10 minutes until the temperature becomes constant. Use the table below for recording data. The minimum temperature reached is the final temperature (T_f). (*Note: One partner should be holding the cup and stirring, while the other is taking the time-temperature readings.*)	5. Final temperature, T_f (°C):
6. Discard the mixture by dumping it into the sink. Rinse the Styrofoam cup thoroughly and wipe dry.	

Time (s)	Temperature (°C)	Time (s)	Temperature (°C)

Part 2: Dissolution of calcium chloride

1. Obtain two Styrofoam cups. Construct a simple calorimeter by nesting one cup inside the other. Weigh the cups. Record exact mass.	1. Mass of cups: _____ grams

2. Measure 100 mL laboratory water using a graduated cylinder, and transfer into the Styrofoam cup. Weigh again. Record exact mass.	2. Mass of water + cups: _____ grams
3. Cover cup with a lid with two holes – one for inserting a thermometer and the other for a stirring rod. Record the temperature of the water to ±0.1^{o}C. This is the initial temperature (T_i).	3. Initial temperature, Ti (^{o}C):
4. Using the tare function, weigh about 20 grams of solid calcium chloride dihydrate ($CaCl_2 \times 2H_2O$) on a weighing paper/boat. Record the exact mass.	4. Mass of $CaCl_2 \times 2H_2O$: _____ grams
5. Transfer the calcium chloride into the Styrofoam cup. Put the lid back, quickly insert the thermometer and stirring rod, and stir the mixture vigorously. Be careful not to hit the thermometer while stirring. Immediately record the temperature of the mixture to ±0.1^{o}C. Continue stirring. Record the temperature of the water at 30 second intervals for 10 minutes until the temperature becomes constant. Use the table below for recording data. The maximum temperature reached is the final temperature (T_f). (*Note: One partner should be holding the cup and stirring, while the other is taking the time-temperature readings.*)	5. Final temperature, T_f (^{o}C):
6. Discard the mixture by dumping it into the sink. Rinse the Styrofoam cup thoroughly and wipe dry.	

Time (s)	**Temperature (^{o}C)**	**Time (s)**	**Temperature (^{o}C)**

Section 4: Calculations

SHOW YOUR WORK on the space provided below.

Part 1: Calculating the heat involved in the dissolution of ammonium chloride

1. Calculate the mass of the solution [(**b**) + (**d**) – (**a**)], in grams.	Mass of the solution (g):
2. Calculate the change in temperature of the solution, $\Delta T = T_f - T_i$, [(e) – (c)]	ΔT (^{o}C):

3. Assuming that the **specific heat of the solution is 4.18 J/g-^{o}C**, calculate the amount of heat involved in dissolving ammonium chloride. *Is this heat lost or heat gained?*	Amount of heat involved, (J):
4. Calculate the number of moles of ammonium chloride used.	Moles of NH_4Cl:
5. Calculate the 'heat of solution' of NH_4Cl (ΔH_{soln}) in kJ/mol. Make sure to include the appropriate sign (+ or -).	(ΔH_{soln}) in kJ/mol:

Part 2: Calculating the heat involved in the dissolution of calcium chloride

1. Calculate the mass of the solution [(**b) + (d) – (a**)], in grams.	Mass of the solution (g):
2. Calculate the change in temperature of the solution, $\Delta \mathbf{T = T_f - T_i}$, **[(e) – (c)]**	ΔT (^{o}C):
3. Assuming that the **specific heat of the solution is 4.18 J/g-^{o}C**, calculate the amount of heat involved in dissolving calcium chloride. Is this *heat lost or heat gained*?	Amount of heat involved, (J):
4. Calculate the number of moles of calcium chloride used.	Moles of $CaCl_2$:
5. Calculate the 'heat of solution' of $CaCl_2$ (ΔH_{soln}) in kJ/mol. Make sure to include the appropriate sign (+ or -).	(ΔH_{soln}) in kJ/mol:

Post Lab Questions:

1. Which dissolution process is exothermic, and which is endothermic? Explain.
2. Compare your calculated ΔH_{soln} values with your classmates. How will you explain the difference in your values? Discuss some possible sources of deviation.

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1.19: Experiment_620_Calculation of the Ideal Gas Constant_1_1_3

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 620: Calculation of the Ideal Gas Constant**

Experiment 620: Calculation of the Ideal Gas Constant

Section 1: Purpose and Summary

Determine the value of the ideal gas constant, R, by measuring the volume, temperature, and pressure of a known amount of gas.

Under ordinary conditions, the pressure (P) of any sample of an ideal gas is inversely proportional to its volume (V) and directly proportional to its absolute temperature (T) and to the number of moles (n) of the gas present in the sample. This proportionality may be written:

$\mathrm{P} \alpha \frac{\mathrm{nT}}{\mathrm{V}}$

where α is read "is proportional to." It is more convenient to put this relationship in the form of an equation:

$P = \frac{nRT}{V}$

where R, the “proportionality constant” is usually called the “ideal gas constant.” This equation is more familiar in the rearranged form PV = nRT, and this equation is called the “ideal gas equation” or the “ideal gas law.”

The purpose of this experiment is to verify this equation for a sample of hydrogen gas, H_2 (g). To do this, we must measure P, V, T, and n. The value of R can then be calculated and compared with the accepted value of R. In this experiment, P, V, and T will be measured directly, while n will be calculated from the amount of magnesium used to produce the hydrogen gas from reacting with hydrochloric acid according to the equation:

Mg(s) + 2 HCl (*aq*) → $MgCl_2$ (*aq*) + H_2 (*g*)

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Wear your safety goggles.

Use caution when handling 6 M HCl. It is corrosive and can burn your skin. If any HCl comes into contact with your skin, rinse it off immediately and thoroughly with lots of water.

Confirm that access to an eyewash station is available and know where it is. 6 M HCl is an eye hazard.

Waste Disposal:

At the end of the experiment, the HCl solution will be much more dilute. The water, diluted HCl and $MgCl_2$ mixture may be rinsed down the sink with plenty of water.

Section 3: Procedure

1. Obtain a piece of magnesium ribbon approximately 5 cm long. Clean the ribbon with fine steel wool and weigh it accurately (to the nearest 0.0001 g). Make sure that it weighs less than 0.04 grams. (If it weighs more, cut off a small piece and re-weigh.). Record the mass of the magnesium strip.	Mass of the magnesium strip: (a) Trial 1: _____ grams Trial 2: _____ grams
2. Roll the ribbon loosely and then wrap it in a little ball of fine copper wire (see the display in the laboratory), leaving a "handle" of copper wire. The wrapping is designed to prevent small pieces of magnesium from breaking off and escaping during the experiment. Make sure that the ball is not too large to fit into the gas measuring tube.	
3. Set up a ring stand with a burette clamp in position to hold a 50-mL gas-measuring tube (eudiometer). Fill a 400-mL beaker about 2/3 full of tap water and place it near the ring stand.	
4. Tilt the gas-measuring tube slightly and pour in about 10 mL of 6 M HCl. (Estimate volume using the marks on the tube, and don't worry about getting exactly 10 mL) Then, with the tube still in the same tilted position, gently add some water from a wash bottle, being careful not to mix the water too much with the acid. Then, gently fill the tube to the top with water (pour from a beaker or wash bottle). While pouring, rinse down any acid that may have wet the sides of the tube. The object is to have acid at the bottom of the tube and water at the top. Try to avoid stirring up the acid layer at the bottom of the tube. Air bubbles that may cling to the insides of the tube can be dislodged by gentle tapping of the tube.	
5. Holding the copper coil by the handle, insert the cage about 3 cm down into the tube. Hook the wire over the edge of the tube where it will be pinched by the rubber stopper and held in place. When you insert the stopper, don't put your finger over the hole in the stopper. Let the water overflow as you insert the stopper so that there are absolutely no air bubbles trapped in the tube.	
6. Add some more water to the hole in the stopper so that it is completely filled with water. Cover the hole in the stopper with your finger and invert the tube in the beaker of water, so that the stoppered end is under water. Once the hole is under water, you can remove your finger; the water cannot now run out. Clamp the tube in place. The acid, being denser than the water, will diffuse down through the water and soon reach the metal sample. When the reaction begins, you will see bubbles of hydrogen gas form.	
7. After the bubbles stop forming, you know that the reaction is completed, but you should wait for a few minutes for the tube to come to room temperature and for bubbles that may be clinging to the sides of the tube to be dislodged. (Tap, if necessary.)	

8. Read and record the volume of the hydrogen gas in the eudiometer to the nearest 0.01 mL.	Volume of the hydrogen gas in the eudiometer: (b) Trial 1: _____ mL Trial 2: _____ mL
9. Without changing the position of the eudiometer, hold a ruler at the top surface of the water level inside the beaker, and measure the distance from the water level in the beaker to the water level inside the eudiometer. Record this height (in millimeters). This will be the "height of the liquid column" referred to in the calculations.	Height of the liquid column: (c) Trial 1: _____ mm Trial 2: _____ mm
10. Measure and record the temperature of the gas (to the nearest 0.1°C) by holding a thermometer against the eudiometer where it contains gas.	Temperature of the hydrogen gas: (d) Trial 1: _____ ^{o}C Trial 2: _____ ^{o}C
11. Record the atmospheric pressure (to the nearest 0.1 mmHg) (the instructor will read the barometer and write today's atmospheric pressure on the board).	Atmospheric pressure: (e) Trial 1: _____ mm Hg Trial 2: _____ mm Hg

12. Disassemble the apparatus and pour the solutions from the beaker and eudiometer down the drain. Rinse the eudiometer well with laboratory water.	
13. Repeat the entire procedure with a second sample of magnesium.	

Section 4: Calculations

For each sample of H_2 gas you collected in Part 1, you collected enough data to determine its pressure, volume, number of moles, and temperature. When you substitute your values of P, V, n, and T into the ideal gas law, PV = nRT, you will be able to determine your own value of R, the ideal gas constant.

	Trial 1	**Trial 2**
1. *Number of Moles (n):* From the mass of magnesium used and the balanced equation, calculate the number of moles of hydrogen gas expected for each trial. Show your equation here:	(f) *n* = _____ mol H_2	(f) *n* = _____ mol H_2
2. Pressure (P): To calculate the partial pressure of hydrogen in the tube, there are two corrections to take into account. The first correction has to do with the fact that the hydrogen gas was collected over water. The gas in the eudiometer is really a mixture of hydrogen gas and water vapor. The contribution of water to the total pressure is called the vapor pressure of water. It depends on temperature and can be looked up in a chemistry handbook. To find the vapor pressure of water in your experiment, look up "water, vapor pressure" in the CRC Handbook of Chemistry and Physics. There will be a table that shows the vapor pressure of water (in mmHg) for any given temperature (in °C). The second correction has to do with the difference in water levels in the eudiometer and in the beaker. If the levels were equal, then the pressure inside and outside would be equal. When the levels are unequal, it means that the atmosphere and the gas inside the eudiometer are not pushing down equally on the surface of the water. The difference in water levels that you measured corresponds to the pressure difference in units of mmH_2O. This can be converted to mmHg, using the relation 13.6 mmH_2O = 1 mmHg: P_{corr} = **(c)** x $\frac{1\ mmHg}{13.6\ mmH_2O}$ Taking both corrections into account: $P(H_2)$ = **(e) – (g) – (h)** Convert the pressure of H_2 to atm.	(g) PH_2O = _____ mmHg (h) P_{corr} = _____ mmHg (i) $P(H_2)$ = _____ atm	(g) PH_2O = _____ mmHg (h) P_{corr} = _____ mmHg (i) $P(H_2)$ = _____ atm
3. *Volume (V):* For each trial, convert the volume of gas (b) from mL to L.	(j) $V(H_2)$ = _____ mL	(j) $V(H_2)$ = _____ mL

4. *Temperature (T):* For each trial, convert the temperature *(d)*, from °C to K.	(k) $T(H_2)$ = _____ K	(k) $T(H_2)$ = _____ K
5. Using the Ideal Gas Law, PV = nRT, calculate an experimental value for R for each trial. R = $\frac{PV}{nT} = \frac{(i)x(j)}{(f)x(k)}$	R_{exptl} = _____	R_{exptl} = _____
6. Report the value of R obtained for each trial and the average value. $R_{\text{average}} = \frac{R_{\text{trial 1}} + R_{\text{trial 2}}}{2}$	$R_{average}$ = _____	
7. Calculate the percent error using the theoretical value of R (0.08206 L•atm/K•mol) and your average experimental value of R. error $\% = \frac{\lvert R_{\text{theoretical}} - R_{\text{average}} \rvert}{R_{\text{theoretical}}} \times 100$	% error = _____	

Show ALL work here:

Post Lab Questions:

1. Why don't you need to measure the exact volume of acid used?

2. In a similar experiment, a piece of aluminum was reacted with HCl. The hydrogen gas produced was collected in a eudiometer in the same way.

a) Write and balance the equation for this reaction. (Hint: what is the formula of aluminum chloride, the other product?)

b) If 39.5 mL of H_2 are produced at 21°C when the atmospheric pressure is 762.8 mmHg, and if the height of the liquid column in the eudiometer is 11.2 cm, what mass of aluminum was used?

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1.20: Experiment_621_Lewis Structures and Molecular Geometry_1_1_4

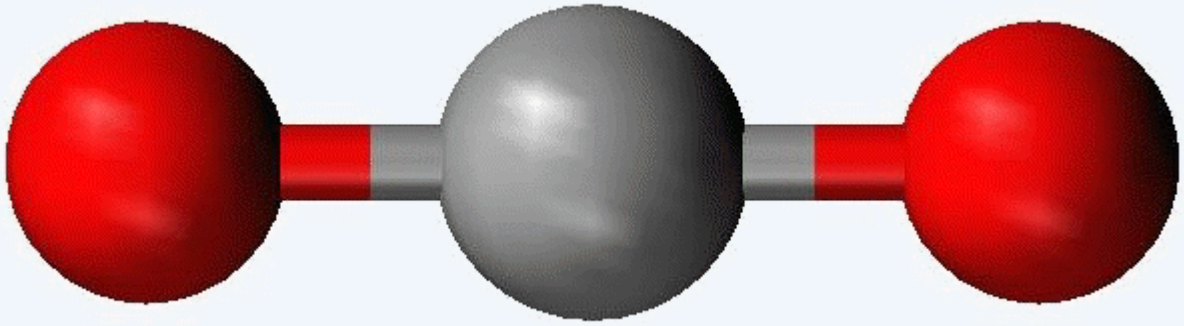
Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 621: Lewis Structures and Molecular Geometry**

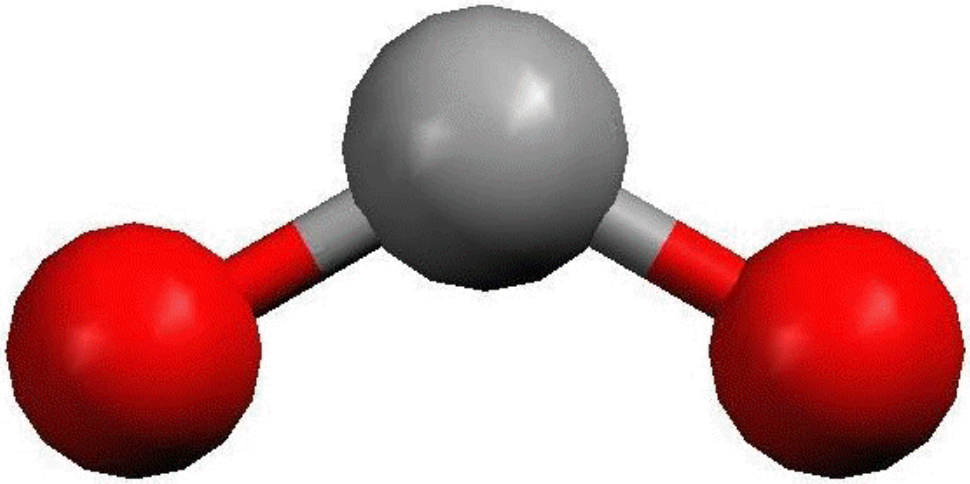
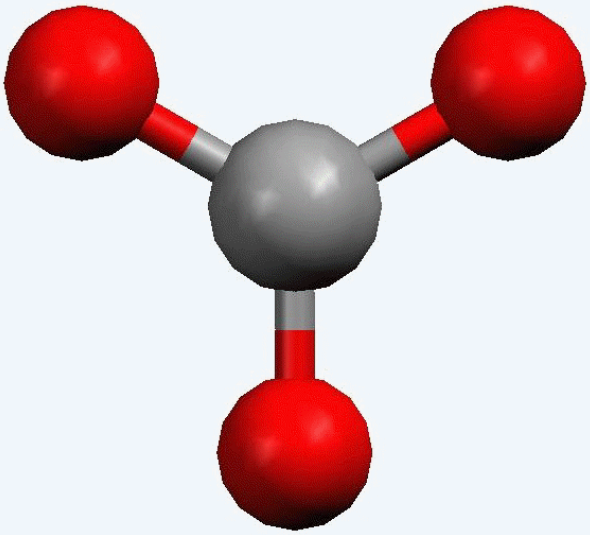
Experiment 621: Lewis Structures and Molecular Geometry

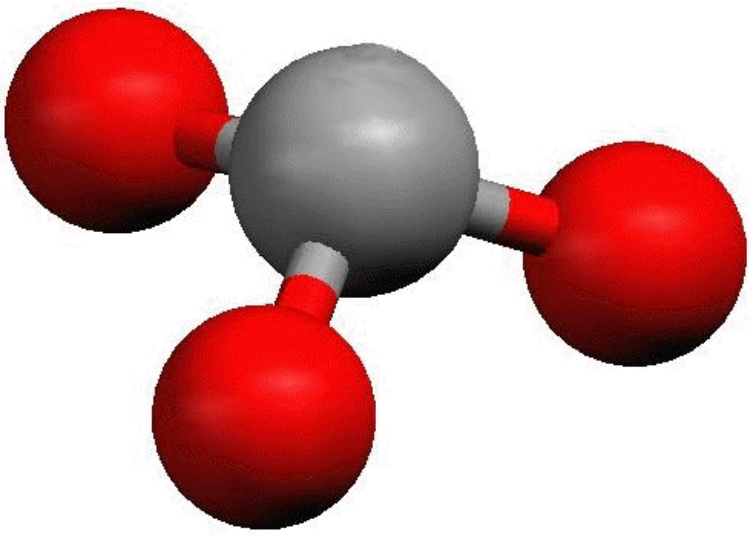
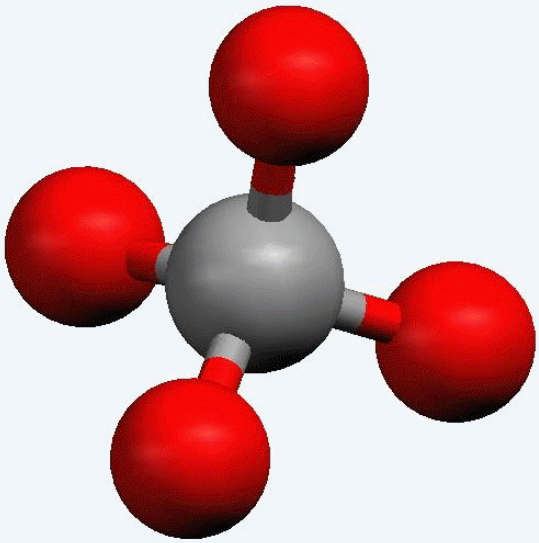
Section 1: Purpose and Summary

- Draw Lewis structures of covalent compounds and polyatomic ions.
- Use a molecular model kit to construct these molecules/ions and visualize their structure and geometry
- Apply VSEPR theory to determine the molecular geometry of a molecule or ion.
- Determine the polarity of a molecule.

Lewis structures are two-dimensional representations of the relative position of atoms in a molecule or polyatomic ion. The Valence Shell Electron Pair Repulsion (VSEPR) theory can then be applied to predict the three-dimensional shape of these molecules or ions. In this experiment, students will practice drawing Lewis structures of covalent compounds and polyatomic ions. Using a molecular model kit, students will build these molecules and determine their shapes and bond angles. The following table may be used as reference:

3D representation	**Mole Shap**
	Linea

	Bent
	Trigo plana

	Trigo pyrar
	Tetra

The difference in electronegativity of the atoms in a bond determine whether a bond is polar or nonpolar. Depending on the arrangement of the polar bonds in a three-dimensional molecular structure, the molecule can be classified as polar or nonpolar. In this experiment, students will also determine the polarity of the molecule based on the structure that they have drawn and modeled.

Section 2: Procedure

Part 1: Drawing Lewis structures

1. Calculate the total number of valence electrons of all atoms in the molecule or ion. For a polyatomic ion, add one electron for each negative charge, and subtract one for each positive charge.

2. Look up and compare the electronegativities of the atoms. The least electronegative atom is the central atom, except hydrogen. Connect the other atoms to the central atom with a line (single bond).

3. Add lone pairs so that each atom is surrounded by eight electrons (**octet rule**). Count 2 electrons for a single bond.

4. Count the total number of electrons in the structure. If the structure contains more electrons than the total number of valence electrons calculated in step 1, form double bonds or triple bonds on the central atom as needed.

Part 2: Constructing molecular models, and determining molecular shapes

1. Use your molecular model kit to construct a three-dimensional model of each of the molecules and polyatomic ions. Sketch a 3D representation of your model.

The following is a quick guide to using your molecular model kit:

- Each colored ball corresponds to a specific atom. Refer to the color key provided in the kit.
- Use the medium length gray links for single bonds.
- Use the short white links for lone pairs.
- Use the flexible long gray links for making double or triple bonds.

2. Compare each model to the list of molecular shapes and identify the shape of the molecule. Determine the bond angle(s).

Part 3: Determining the polarity of the molecule

Using electronegativity values, evaluate the polarity of the bonds in each molecule. A bond is considered polar if the electronegativity difference between the bonded atoms is 0.5 to 0.9. A bond is considered nonpolar if the electronegativity difference between the bonded atoms is less than 0.5.

- A molecule will be **nonpolar** if it contains all nonpolar bonds, OR it contains polar bonds that are arranged in a symmetrical manner around the central atom.
- A molecule will be **polar** if it contains polar bonds that are arranged in a nonsymmetrical manner resulting from the presence of lone pairs on the central atom, or when there are different atoms surrounding the central atom

Section 3: Worksheet

1. **CH_4**

Total number of valence electrons:	Lewis structure:
3D sketch:	Molecular shape / bond angle Polar or nonpolar?

2. **CO_2**

Total number of valence electrons:	Lewis structure:
3D sketch:	Molecular shape / bond angle Polar or nonpolar?

3. **NH_3**

Total number of valence electrons:	Lewis structure:
3D sketch:	Molecular shape / bond angle Polar or nonpolar?

Section 3: Worksheet (continued)

4. **H_2O**

Total number of valence electrons:	Lewis structure:
3D sketch:	Molecular shape / bond angle Polar or nonpolar?

5. **N_2**

Total number of valence electrons:	Lewis structure:
3D sketch:	Molecular shape / bond angle Polar or nonpolar?

6. **SO_2**

Total number of valence electrons:	Lewis structure:
3D sketch:	Molecular shape / bond angle Polar or nonpolar?

Section 3: Worksheet (continued)

7. **O_2**

Total number of valence electrons:	Lewis structure:
3D sketch:	Molecular shape / bond ang Polar or nonpolar?

8. **O_3 - use yellow ball for central atom**

Total number of valence electrons:	Lewis structure:
3D sketch:	Molecular shape / bond angl Polar or nonpolar?

9. **CO**

Total number of valence electrons:	Lewis structure:
3D sketch:	Molecular shape / bond ang Polar or nonpolar?

Section 3: Worksheet (continued)

10. **CO_3^{2-}**

Total number of valence electrons:	Lewis structure:
3D sketch:	Molecular shape / bond ar Polar or nonpolar?

11. **NO_3-**

Total number of valence electrons:	Lewis structure:

3D sketch:	Molecular shape / bond ar Polar or nonpolar?

12. **CF_2Cl_2**

Total number of valence electrons:	Lewis structure:
3D sketch:	Molecular shape / bond Polar or nonpolar?

Post Lab Questions:

1. Which of the above molecules/ions has (have) more than one equivalent Lewis structures?
2. Which of the above molecular structures contain polar bonds, and yet the molecule is nonpolar? Explain briefly.

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1.21: Experiment_622_Electrical Conductivity_1_1_3

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 622: Electrical Conductivity of Aqueous Solutions**

Experiment 622: Electrical Conductivity of Aqueous Solutions

Section 1: Purpose and Summary

- Observe the electrical conductivity of different substances in aqueous solutions.
- Determine whether the solution is a strong electrolyte, weak electrolyte, or nonelectrolyte.

The ability of a substance to conduct electric current is due to the movement or migration of ions in solution. Substances that are completely ionized in solutions are highly conducting and thus called strong electrolytes. Examples include salts, strong acids, and strong bases. Partially ionized substances, on the other hand, are weakly conducting and thus called weak electrolytes. Substances that do not ionize in solution are nonelectrolytes.

In this experiment, students will determine the conductivity of different aqueous solutions using a conductivity tester. Based on the results, students will classify each solution as strong electrolyte, weak electrolyte, or nonelectrolyte.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Be careful when handling acid solutions. Even at low concentrations, acids may irritate your skin.

When using the conductivity tester, do not immerse the circuit board to water. Only the copper electrodes (metal probes) should be immersed and rinsed with laboratory water.

Waste Disposal:

While you are doing the experiment, collect your used aqueous solutions into a waste beaker.

When you are finished with the experiment, pour the contents of the waste beaker into the inorganic waste container in the fume hood.

Section 3: Procedure

Conductivity Testing – Evidence for Ions in Aqueous Solutions

Simple conductivity testers have a 9V battery and two parallel copper electrodes. Different styles of conductivity meters exist. The following is for a conductivity meter with a green and a red light-emitting diode (LED). **Refer to the operating instructions on the specific use of your conductivity meter and adapt this procedure appropriately.**

Use a wash bottle containing laboratory water and a ‘waste’ beaker to rinse the copper electrodes. Dry the copper electrodes completely with Kimwipes™ tissue.

When switched on, and the copper electrodes are dipped into a conducting solution, the LEDs will glow.

The photo shows the conductivity meter in a small beaker. Note that the electrodes are just submerged below the level of the liquid. The rest of the circuit board and battery power supply are kept dry.

It is very important to avoid contaminating solutions when moving the electrodes from one to another solution. It is a good idea to keep a separate container with laboratory water to use as a rinse solution between testing solutions. If you do this, change the laboratory water frequently.

The following key can be used to compare the conductivity of the solutions. This key is from the model which has a red and green LED. Other keys may need to be developed for other meters.

Scale	Red LED	Green LED	Conductivity
0	Off	Off	None
1	Dim	Off	Low
2	Medium	Off	Medium
3	Bright	Dim	High
4	Very bright/Blinking	Medium	Very high

When testing samples/solutions, switch the conductivity tester on and dip the copper electrodes into the sample/solution.

After each test, switch the tester off, rinse the electrodes thoroughly with laboratory water, and dry with Kimwipes™. In addition, dispose the sample/solution and rinse the beaker thoroughly between tests.

Record your results on the table following this procedure.

1. **Laboratory water vs. tap water**

1. Pour 5 mL of laboratory water into a clean, dry 50-mL beaker. Test the conductivity and record your results.
2. Repeat this step, using 5 mL of tap water in place of the laboratory water.

2. **Solid vs. aqueous solution of a compound**

1. Transfer about 0.2 g of solid sodium chloride (NaCl) into a clean, dry 50-mL beaker. Test the conductivity and record your results. Be sure the NaCl covers the electrodes.
2. Into the same beaker, add 5 mL of laboratory water to dissolve the salt. Test the conductivity and record your results.
3. Repeat steps (a) and (b), using calcium carbonate ($CaCO_3$) in place of NaCl.

3. **Conductivity of various aqueous solutions**

1. Pour 5 mL of each of the solutions listed in the data table into a clean, dry 50-mL beaker. Test the conductivity and record your results.
2. Make sure to rinse the conductivity tester and beaker in between tests.

Sample/Solution	**Observations**		**Conductivity**
	Red LED	**Green LED**	
Examples:	Bright	Dim	High
LiOH(aq)	Dim	Off	Low
HNO_2(aq)	Off	Off	None
Methanol, CH_3OH(l)			

Sample/Solution	**Observations**	
	Red LED	**Green LED**
Laboratory water		
Tap water		
NaCl(s)		
NaCl(aq)		

$CaCO_3(s)$		
$CaCO_3(aq)$		
0.1 M Acetic acid, $HC_2H_3O_2(aq)$		
“Glacial” Acetic acid, $HC_2H_3O_2$		
0.1 M $Al(NO_3)_3(aq)$		
15 M $NH_3(aq)$		
0.1 M $Ca(OH)_2(aq)$		
Ethanol, $C_2H_5OH(l)$		
0.1 M HCl(aq)		
$Mg(OH)_2$ (sat. aq)		
0.1 M $MgSO_4(aq)$		
0.1 M $HNO_3(aq)$		
0.1 M KI(aq)		
0.1 M NaOH(aq)		
Sparkling water		
Sucrose, $C_{12}H_{22}O_{11}(aq)$		

Section 4: Data Analysis

Based on your observations, classify each sample as strong electrolyte, weak electrolyte, or nonelectrolyte. List them in the table

below

Strong electrolytes	Weak electrolytes	Nonelectrolytes

Post Lab Questions:

1. Why must the copper electrodes in the conductivity tester, as well as the beaker, be rinsed with laboratory water after each test?
2. Was there a difference in conductivity between laboratory water and tap water? Why do you think this is so?
3. Was there a difference in conductivity between solid NaCl and aqueous NaCl? Why do you think this is so?
4. Based on your results, among the tested substances, which are the:

1. Strong acids
2. Weak acids
3. Strong bases
4. Weak bases

5. Write the chemical equations for the complete dissociation of at least three strong electrolytes listed in your data table in Section 4.

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1.22: Experiment_623 Estimating the Calorie Content of Wax 1_3

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 623: Estimating the Calorie Content of Wax**

Experiment 623: Estimating the Calorie Content of Wax

Section 1: Purpose and Summary

- Understand the general chemical formula for wax
- Set up an apparatus to measure the amount of heat generated from combustion of wax
- Calculate the calorie content of wax

Alkanes are saturated hydrocarbons. They contain only carbon and hydrogen and have no multiple (double or triple) bonds present. Low molecular weight hydrocarbons are gases like methane and ethane. Medium molecular weight hydrocarbons are found in liquids like gasoline or diesel fuels. High molecular weight hydrocarbons are found in tar and waxes.

When hydrocarbons are burned with oxygen in a combustion reaction, the reaction releases heat. A general reaction equation is:

$2C_nH_{2n+2} + (3n+1)\ O_2 \rightarrow 2n\ CO_2 + (2n+2)\ H_2O$

Reactants Products

From the standard heats of formation of the substances involved, the heat of any reaction ($\Delta H^o_{reaction}$) can be determined from the equation:

$\Delta H^o_{reaction} = \sum \Delta H^o_f$, products - $\sum \Delta H^o_f$, reactants

The ΔH^o_f (heats of formation) from published tables (NIST Webbook, CRC Handbook of Chemistry and Physics, and the like) can be used for this calculation. Note that when the $\Delta H^o_{reaction}$ is positive, heat is absorbed, and when the $\Delta H^o_{reaction}$ is negative, heat is released.

Several different sets of units can be used to measure $\Delta H_{reaction}$. We will be using calories per gram and calories per mole. “Nutritional calories” that are declared on food packaging are kilocalories (kcal), or 1000 calories. So a candy bar that is listed as 240 calories means: 240,000 calories of energy are available by metabolizing it.

Candle wax has carbon chains that are very long, generally at least 20 or 30 carbons bound together in long chains. For this combustion, if a value of n = 25 is used, the balanced equation becomes:

$C_{25}H_{52} + 38\ O_2 \rightarrow 25\ CO_2 + 26\ H_2O$

In this experiment, students will use a measured amount of water to absorb the heat from a lighted candle. By determining the increase in temperature of the water, students can calculate how much heat has been absorbed by the water and therefore, how much has been released from burning the candle.

If we assume that the amount of heat energy, **q**, from combustion is transferred from the flame of the candle through the aluminum can and into the water suspended above the candle, then we can describe it by the following relationship:

heat absorbed by the water = heat given off by the candle

= (mass)(specific heat)(change in temperature)

$q = m \times c \times \Delta T$

Be careful with your units and unit conversions. Heat is usually given in either calories or joules, mass is in grams, the specific heat is in calories per gram degree C, and change in temperature is in degrees C.

If the candle is weighed before and after heating, students can measure the mass lost during the combustion. This will allow for the calculation of the heat generated per unit mass of candle wax combusted.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Waste Disposal:

The aluminum cans be reused. The burned candle can be disposed of in the garbage bin.

Section 3: Procedure

1.  Obtain a clean, empty, dry aluminum can. Measure the mass of the can to the nearest 0.01 g and record this value. Using a thumbtack, pierce the can about 1/3 height of the can in four spots. Open up a large paperclip and run the paperclip through the can from one side to the other. Repeat with a second paperclip. You should now be able to suspend the can from an iron ring by the paperclips. See the photo to the left.	Mass of aluminum can: 1. _____ grams
2. Place the can on a balance that measures to the nearest 0.01 g. Tare the balance. Add approximately 100 ml of laboratory water to the can and measure the mass of water added. Record this value.	Mass of aluminum can and laboratory water: 2. _____ grams
3. Obtain a candle and measure its mass to the nearest 0.001 g.	Initial mass of candle: 3. _____ grams

4. Measure the initial temperature of the water in the can to the nearest 0.1 degree Celsius. Either a glass themometer or a digital thermometer may be used.	Initial temperature of the water: 4. _____ ^{o}C
5.  Place the candle under the can. Wrap with aluminum foil the lower half of the can and the space between the can and the base of the candle around the can (see right). (The candle will not remain lit if you completely surround the space.) Light the candle and heat the can directly with the flame. Monitor the temperature until there is an increase in the temperature of the water of 10 degrees Celsius. This should take less than 10 minutes. Extinguish the candle and record the final temperature of the water in the can.	Final temperature of the water: 5. _____ ^{o}C
6. Measure the final mass of the candle to the nearest 0.001g.	Final mass of candle: 6. _____ grams
7. Repeat this procedure for a second measurement. If time permits, perform a third measurement. Tabulate your data in the worksheet below.	

Section 4: Calculations and Tabulation of Data

1. Calculate the mass of the laboratory water in the can: [(**b**) – (**a**)], in grams.	**Mass of water (g):**
2. Calculate the change in temperature of the water, $\Delta T = T_f - T_i$, **[(e) – (d)]**	**ΔT (^{o}C):**
3. Using the specific heat of water, **1.00 cal/g-^{o}C**, calculate the amount of heat absorbed by the water from the burning candle.	**Amount of heat absorbed by the water, (cal):**
4. Calculate the mass of the burned candle: **[(f) – (c)]**	**Mass of burned candle (g):**
5. Calculate the calorie content of the wax from this candle, in cal/g. Make sure to include the appropriate sign (+ or -).	**Calorie content of wax, cal/g:**

6. Determine the average calorie content of wax from your result and compare with the class.	

Data Summary:

	Trial 1	**Trial 2**	**Trial 3**
Mass of water, g			
Change of temperature of water, ^{o}C			
Amount of heat absorbed by the water, cal			
Mass of burned candle, g			
Calorie content of wax, cal/g			
Average calorie content of wax, cal/g			
Average value for the class			

Post Lab Questions:

1. Many tables use values of joules per mole, or kilojoules per mole. Using a chemical formula of $C_{25}H_{52}$ for wax, convert your value of the calorie content per gram into the units of calories per mole, kilocalories per mole and kilojoules per mole.

2. Using thermodynamic data available, calculate the molar heat of combustion ($\Delta \mathbf{H^{o}_{comb}}$) for the following chemical equation:

$C_{25}H_{52} + 38\ O_2 \rightarrow 25\ CO_2 + 26\ H_2O$

3. How does the class average calorie content of wax compare to the calculated value above (2).

4. Aluminum has a specific heat of 0.215 cal / g °C . Calculate the amount of heat that is absorbed by the can for one of your trials. Is this a significant amount relative to the amount of heat absorbed by the water? If it is, correct your three trials.

Notes:

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1.23: Experiment_624_Measuring pH_1_2

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 624: Measuring pH**

Experiment 624: Measuring pH

Section 1: Purpose and Summary

- Determine the pH of some solutions using 'red cabbage' indicator.
- Determine the pH of solutions using a pH meter.
- Understand the effect of adding an acid or a base to buffer solutions.

In this experiment, the instructor will prepare a pH indicator by extracting colored substances from red cabbage. This pH indicator solution will be added to buffer solutions of pH range 1 – 13. Students will record the resultant color of each buffer solution. Students will then use the indicator to determine the pH of common solutions by comparing the color to the buffers. To measure the exact pH values of solutions, a laboratory pH meter will be used. Students will determine the hydrogen ion concentration, [H+] of the solutions from the pH value recorded. Students will determine the effect of adding HCl or NaOH to an acetic acid/acetate buffer solution.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Waste Disposal:

All reagents used in this experiment are generally non-toxic and may be disposed of in sinks with tap water rinses.

Section 3: Procedure

Part 1: Preparing the pH indicator and pH standards – INSTRUCTOR DEMO

NOTE: Part 1 may be replaced by the use of universal indicating solution at the instructor's option.

1. Place several cut-up red cabbage leaves into a 600-mL beaker and cover the leaves with laboratory water.
2. Boil the cabbage leaves on a hot plate until the pigment has been extracted. The leaves will look whitish in color.
3. While the indicator cools, prepare an array of buffer solutions with pH 1 – 13. Label thirteen (13) 150-mm test tubes. Fill each test tube with about a half-full of the appropriate buffer solution.

4. Using a dropper pipet, add several drops of the cabbage indicator to each test tube. An array of colors should be observed. Students will record the color of each pH standard.

Buffer pH	Color with Cabbage Indicator
1	
2	
3	
4	
5	
6	
7	
8	
9	
10	
11	
12	
13	

Part 2: pH determination of household products or everyday chemicals

Possible household items: white vinegar, colorless soda like Sprite™, dish soap solution (colorless), sparkling water, alkaline water, isopropyl alcohol

Everyday chemicals: 0.1 M HCl, 0.1 M acetic acid, 0.1 M ammonia, 0.1 M NaOH

1. Take a 150-mL beaker and obtain about 50 mL of cabbage indicator solution prepared in Part 1.
2. Label ten (10) clean medium test tubes with the solutions to be tested.
3. Transfer about 3 mL of each solution into the corresponding test tube.
4. Using a dropper pipet, add an equal volume of cabbage indicator solution. Mix well.
5. Compare the resulting color of the solution with the buffer array to determine the approximate pH of the solution. Record your observations on the table below.
6. Set aside these solutions for use in the following section.

Solutions tested	**Approximate pH**

Part 3: Quantitative determination of pH

This procedure describes how to use a portable Flinn pH meter (model AP8673). If the pH meter is not measuring properly (unstable, or inaccurate), refer to the manual for troubleshooting.

1. Check out a portable pH meter from the stockroom.
2. Remove the protective cap on the electrode. Clean any salt build-up off by rinsing with laboratory water.
3. Press the ON/OFF button once.
4. Rinse the electrode with laboratory water and blot dry with filter paper.
5. Take one of the solutions from Part 2 of this experiment.
6. Immerse the electrode in the test solution. Once the display stabilizes (approx. 1 min.), record the exact pH.

7. Remove the pH meter from the solution.
8. Repeat steps 4 – 7 using the rest of the test solutions.
9. When finished, rinse the electrode with laboratory water and blot dry with filter paper. Replace cap and return the pH meter to the stockroom.

Solutions tested	**pH**

Part 4: Effect of adding an acid or a base to the pH of a buffer

1. Obtain four (4) medium test tubes and label them A – D.
2. To test tubes A and C, add 10 mL of laboratory water.
3. To test tubes B and D, add 5 mL of 0.1 M acetic acid ($HC_2H_3O_2$) and 5 mL of 0.1 M sodium acetate ($NaC_2H_3O_2$). This mixture is your acetic acid/acetate buffer solution. Stir well to mix.
4. Using Hydrion^{TM} pH paper strips, determine the pH of the contents of test tubes A – D. Put four (small) strips of pH paper on a large watch glass. Using a stirring rod, dab a small drop of the test solution onto the pH paper. Compare the color obtained to the pH scale on the label of the pH paper casing. Record the pH values to the tenth place.
5. To test tubes A and B, add five (5) drops of 0.1 M hydrochloric acid (HCl). Determine the pH using pH (similar to step 4 above).
6. To test tubes C and D, add five (5) drops of 0.1 M sodium hydroxide (NaOH). Determine the pH using pH (similar to step 4 above).

Solutions tested	**pH**	**pH after addition of 0.1 M HCl**
A (laboratory water)		

B (buffer solution)		
Solutions tested	**pH**	**pH after addition of 0.1 M NaOH**
C (laboratory water)		
D (buffer solution)		

Post Lab Questions:

1. Based on your results, classify each test solution as acidic, basic, or neutral.
2. Write the mathematical equation that shows the relationship between pH value and the hydrogen ion concentration, $[H^+]$. What is the unit of $[H^+]$?
3. Using your results in Part 3 of this experiment, calculate $[H^+]$ for each test solution. Include units.
4. Compare a solution with pH 10.5 and a solution with pH 4.5. Which solution contains more H^+ ions? Explain.
5. Two methods were used in determining the pH of the solutions – pH paper and pH meter. Do they show similar or different pH values? What was different?
6. Compare the pH of the buffer solutions in Part 4 of this experiment before and after the addition of 0.1 M HCl and 0.1 M NaOH. Does this confirm the expected behavior of a buffer solution against an added acid or base? Explain.

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1.24: Experiment_625_The Bunsen Burner and Glass Working_1_2

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 625: The Bunsen Burner and Glass Working**

Experiment 625: The Bunsen Burner and Glass Working

Section 1: Purpose and Summary

- Gain familiarity with how to properly use a Bunsen burner to generate heat and open flames.
- Reinforce safe laboratory practices when using gas and open flames.
- Use the Bunsen burner to work with glass tubing: fire polishing, extruding, and bending.

Fire and the ability to generate heat is an important concept both in everyday life as well as in the laboratory. Without fire and heat, we could not cook food or stay warm in the winter. In the laboratory, fire and heat is oftentimes needed to facilitate a chemical reaction or other type of transformation of matter. To generate heat in the laboratory, we can use a Bunsen burner to produce an open flame. This requires a fuel source (gas, often methane) and oxygen (usually from the air around us). To produce an optimal flame, the Bunsen burner needs to be adjusted to produce an efficient mixture of gas and oxygen.

Bunsen burners used in most laboratories are made of metal that consists of a mixing tube (barrel) attached to the base of the Bunsen burner. You can reference “Technique H: Use of a Bunsen burner” for a detailed description and instructions for use. The base of the Bunsen burner has a gas inlet where rubber tubing is used to connect the Bunsen burner to the gas source on the laboratory bench (Fig.1). On the bottom of the Bunsen burner base, there is a needle valve that screws into the bottom of the Bunsen burner base (Fig.2). Adjusting the needle valve allows more or less gas to enter the Bunsen burner that will mix with oxygen in the barrel. To adjust the amount of oxygen, the barrel can be rotated to allow more or less oxygen through the collar. The collar covers the air inlet holes that allow oxygen to enter the barrel (Fig.3). Once an acceptable mixture of gas and oxygen is flowing through the Bunsen burner, the fire needs to be initiated with a spark or flame. This can be accomplished using a striker or a match to introduce a spark or flame at the top of the Bunsen burner barrel. The initial flame produced by the Bunsen burner may not be optimal so the needle valve and barrel will likely need to be adjusted to achieve a more optimal mixture of gas and oxygen.



Figure 1

Gas inlet and rubber tubing.

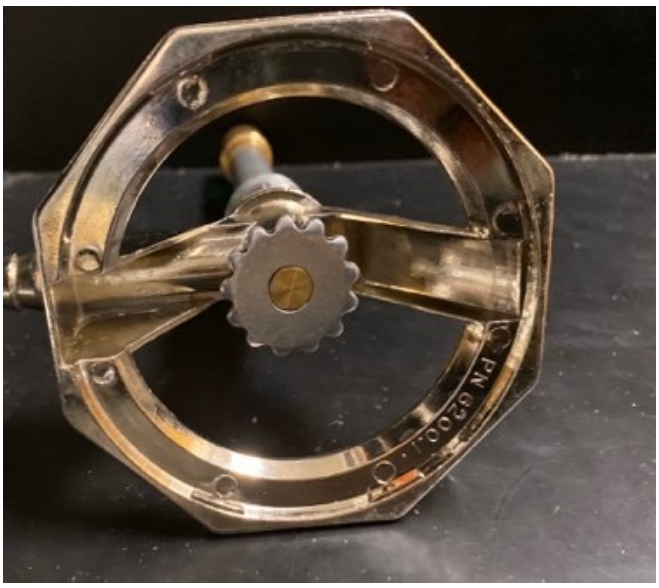


Figure 2

Needle Valve.

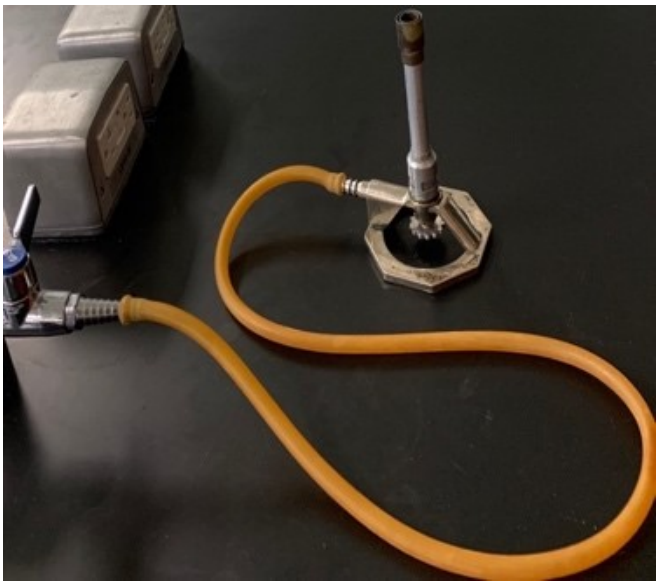


Figure 3

Complete set up of Bunsen Burner.

An optimal flame receiving the right mixture of gas and oxygen typically has an inner blue cone surrounded by an outer translucent (almost invisible) blue flame (See Fig. 5). Sometimes a third colorless cone will appear inside the inner blue cone that is made up of unburned gas. This optimal flame can be called an oxidizing or nonluminous flame. The hottest part of the flame is the upper tip of the inner blue cone (see image). Different locations within an optimal flame have different temperature and heat outputs. A flame that looks orange or yellow and does not have an inner blue cone is receiving too much gas relative to the amount of oxygen. This may be called a luminous or reducing flame or a cool flame.



Figure 4

Example of sparking flame.



Figure 5

Luminous flame.

As previously described, fire is an important and beneficial resource in life. However, uncontrolled fire and fire that ignites in unintended places can be dangerous. Thus, proper procedures and safety precautions should be used when working with fire and open flames. These include the following (in no particular order of importance):

a) A burning fire/flame should always have a person present to monitor and watch that it doesn't become uncontrolled.

b) When working closely to an open flame, we need to make sure not to get too close to the flame so nothing catches fire that we do not want to (hair, clothing, other chemicals or lab materials, etc).

c) Care should be taken to make sure that gas is not on when it is not fueling the lit Bunsen burner. If you smell gas, turn off the main gas source on the laboratory bench and wait a few moments before trying to reignite your Bunsen burner.

d) Make sure the gas is turned off when you are finished with your experiment and ready to leave the laboratory.

e) Parts of the Bunsen burner and items you have heated up can get hot even though you can't see that they're hot. Use precaution when handling the Bunsen burner that was just turned off and other items that you have recently heated.

f) Always wear lab goggles when working in the laboratory and take special attention to make sure your lab goggles are on when you or someone around you is working with an open flame.

In this experiment, students will work with the Bunsen burner to produce an optimal flame. Then, students will use the flame to do somewhat practical applications.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

The materials in this experiment are generally harmless. Use of eye protection is recommended for all experimental procedures.

Tie up or tie back any loose hair. Keep loose fitting clothing away from open flames.

Hot glass looks like cold glass. A good practice is to place the glass you recently heated facing away from you.

Waste Disposal:

Dispose of any unwanted glass in the broken glass container.

PreLab Questions:

1. What safety precautions should you take while doing the following?

1. Lighting the Bunsen burner and adjusting the flame
2. Cutting and fire polishing glass tubing (See Figs. 6-8)
3. Making the dropper tip
4. Bending glass tubing

2. If your Bunsen burner flame is not the right mix of gas and oxygen when you first light it, how do you adjust the Bunsen burner to get to the optimal flame?

3. What does an optimal flame look like? What is the hottest part of the optimal flame?

4. Briefly summarize the procedure used to cut the glass tubing before fire polishing it.

5. What is the purpose of the wing tip that you put on top of the Bunsen burner? What color should the flame be when it is coming out of the wing tip?

6. You get to bend glass to make a shape of your choice from your imagination at the end of this experiment. What ideas do you have for the shape that you want to make by bending glass tubing?

Section 3: Procedure

Part 1: Lighting and Adjusting the Bunsen Burner

Reference “Technique H: Use of a Bunsen burner” for a detailed description and instructions for use.

1. Get a Bunsen burner and a piece of rubber tubing for Bunsen burners. Check for any cracks or damage on the rubber tubing. Get a new piece of tubing if yours is damaged. Attach one end of the tubing to the gas source on the laboratory bench and the other end to the gas inlet on the Bunsen burner.

2. Get a match or striker ready. If using a striker, move the movable arm on the striker so that the piece of flint rubs across the rough surface. This will provide a spark that can be used to ignite the Bunsen burner (see image). Use the striker to produce a spark near the top of the barrel of the Bunsen burner.

3. Turn on the gas source on the laboratory bench. If it’s a knob, turn it counterclockwise to open and clockwise to close. If it’s a lever style, perpendicular to the outlet valve means closed and parallel means open. Light the match or use the striker to produce a spark and bring it near the top of the barrel of the Bunsen burner. This will light your Bunsen burner.

4. Observe the initial flame produced by the Bunsen burner. Record how your initial flame looks below. Is this an optimal flame? Initial flame observation:
5. Get a piece of iron wire to test the relative temperature and heat of different parts of your initial flame. Use a test tube clamp or crucible tongs to hold the iron wire. Put the iron wire into various locations in your initial flame and see what happens. Record your observations below. Iron wire in initial flame observations:
6. Adjust the needle valve and/or the barrel and collar to change the amount of gas and/or oxygen going into the Bunsen burner. You are trying to achieve an optimal flame that is the result of an efficient mixture of gas and oxygen.
7. Hopefully you were able to generate an optimal flame. What does your final flame look like? Final flame observation: What did you have to adjust on your Bunsen burner to get your initial flame to your final flame? How easy or difficult was it to produce an optimal flame? Remarks:
8. Use the iron wire with test tube clamp or crucible tongs again to test various locations in your final flame and see what happens. What happens to the iron wire when you put it in the hottest part of your final flame? Record your observations below. Iron wire in final flame observations:
9. Turn off the gas source at the laboratory bench. This will extinguish your Bunsen burner flame.

Part 2: Fire Polishing and Extruding Glass Tubing

1. Get two (2) pieces of approximately 20 cm long glass tubing, a triangular file, and wire gauze.
2. You will be breaking a piece of glass tubing in half by scoring it then breaking it (See Fig. 6). Place one piece of glass tubing flat on the lab bench. Hold the triangular file so that your thumb or index finger is on a flat edge of the file and the “cornered” edge of the file is pointing down toward the glass tubing. Use a firm, single stroke of the file to score (make a scratch) on the surface of the glass tubing. You should hear a slight grinding sound as the file is scratching the glass. Look for the scratch mark on the glass. If you need to, score the glass again in the same place until you can see a little scratch.
3. Hold the tubing in your hands so the scratch faces away from your body (See Fig. 7). Place each thumb on either side of the scratch mark and break the glass away from you. It may help to wet the scratch mark first with a bit of water. It should not take much pressure from your thumbs if you scored the glass tubing properly.

4. Light your Bunsen burner as you learned in Part 1.

5. Fire polish the newly broken ends of your glass tubing by place the broken end into the hottest part of your Bunsen burner flame. You will likely see the tip of your glass tubing turn orange when the fire polish is complete. Take your fire polished glass tubing out of the flame and place on the wire gauze to cool. Turn off your Bunsen burner. Record any observations below on what you experienced as you broke and fire polished your glass tubing.

Observations (fire polish):

6. You will use the other piece of glass tubing to make dropper tips. Light your Bunsen burner. Hold the glass tubing horizontally so the middle of the glass tubing is in the hottest part of the Bunsen burner flame. Rotate the glass tubing between your index finger and thumb so the glass tubing gets evenly heated on all sides.

7. When the glass tubing turns gets soft and starts to sag (the flame will turn orange and the glass may start to glow orange), pull the ends of the tubing away from each other at a consistent speed.

8. Quickly remove the tubing from the flame and continue to pull the ends of the tubing away from each other. Place the two dropper tips on your wire gauze to cool. Turn off your Bunsen burner.

9. When the glass is cool, use the triangular file to cut the dropper tips to your desired size and open up the end of your dropper tips. Record any observations below on what you experienced as you produced the dropper tips.

Observations (dropper tips):

10. Ask your instructor to observe your fire polished glass tubing and dropper tips and initial below.

Instructor initials:

Fire polish _____ Dropper tips _____

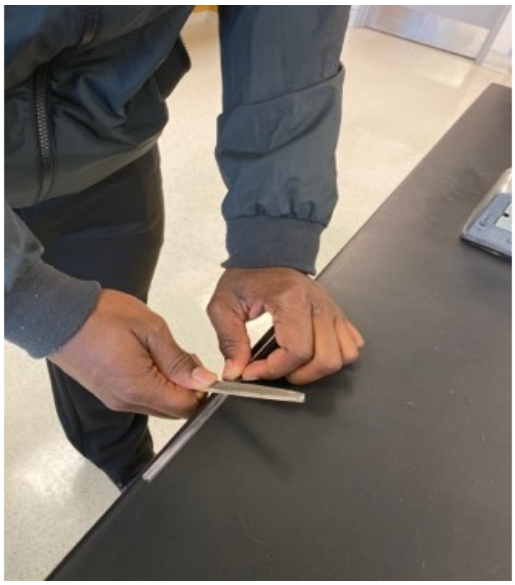


Figure 6

Scoring glass tube.



Figure 7

Proper finger placement to break the glass.



Figure 8

Endpoint of fire polishing edge.

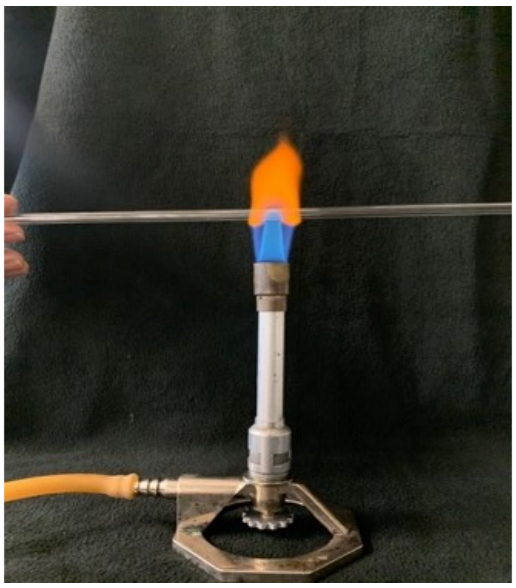


Figure 9

Heating tubing before pulling to form dropper.



Figure 10

Pulling the glass tube apart to form dropper.

Part 3: Bending Glass Tubing

1. Get three(3) pieces of glass tubing and a wing tip.
2. Make sure your Bunsen burner is cool. Then, place the wing tip on the top of the barrel of your Bunsen burner. The wing tip allows you to spread the Bunsen burner flame so you can heat up a wider surface area of the glass tubing.
3. Light your Bunsen burner. The flame should still be blue but now it is spread out instead of looking like a candle flame. You may need to adjust your gas and/or oxygen flow a bit.
4. Hold one piece of glass tubing so the middle is in the Bunsen burner flame. Position the tubing so it is almost parallel with the wing tip and flame. This is heating up a larger surface area of your glass tubing. Rotate the glass tubing so the glass tubing gets evenly heated on all sides.
5. When the glass begins to sag (the flame will turn orange and the glass may start to glow orange), remove the tubing from the flame and bend it to make a 90 degree angle. Your glass tubing should be smooth throughout the bend and the same diameter as the original glass tubing (not kinked). Place the bent glass tubing on your wire gauze to cool. Record any observations below on what you experienced as you produced the 90 degree bend glass tubing. Observations (90 degree bend):
6. Repeat steps 4-5 above with a new piece of glass tubing and make a 135 degree bend glass tubing. Record any observations below on what you experienced as you produced the 135 degree bend glass tubing. Observations (135 degree bend):

7. Repeat steps 4-5 above and your experience using the Bunsen burner so far in this lab to make a new shape from your imagination. Ideas may include a corkscrew, a letter in the alphabet, a small square, etc. Make sure that if your new shape requires bending in multiple places, let the area you just heated and bent cool off before you hold that area with your hands to make the next bend. What shape did you make? Draw it out below and record any observations on what you experienced while making the new shape from your imagination.

Drawing and observations (shape from your imagination):

8. Make sure to turn off your Bunsen burner if you have not already.

9. Ask your instructor to observe your bent glass tubing and shape from your imagination and initial below.

Instructor initials:

90 degree bend _____ 135 degree bend _____

Shape from imagination:

10. Disconnect your Bunsen burner from the lab bench gas supply and return the tubing and other equipment. Discard of any unwanted glass from this experiment in the broken glass container. If you choose to take any glass shapes out of lab with you, make sure you store them in a place where they will not break as you transport them.



Figure 12

Luminous flame

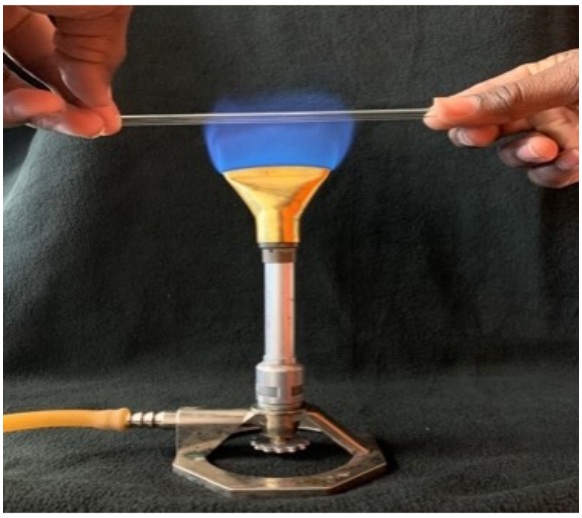


Figure 13

Optimal place to have glass tubing

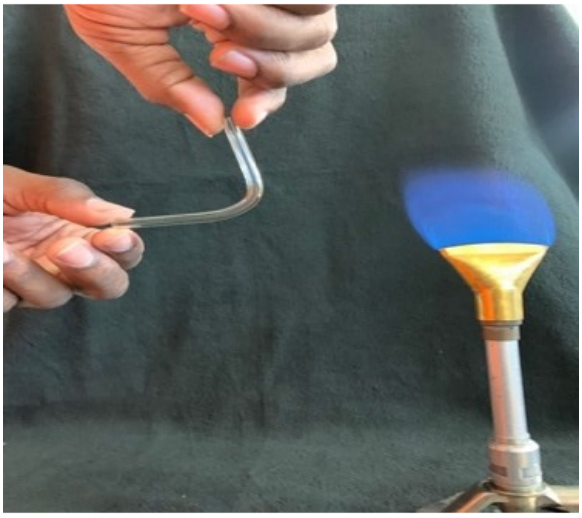


Figure 14

90 degree bend

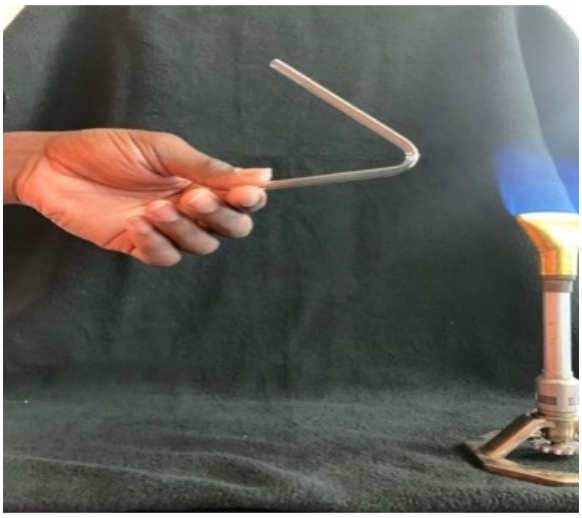


Figure 15

135 degree bend

Notes:

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1.25: Experiment_718_Making Soap - Saponification_1_1_1

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 718: Making Soap - Saponification**

Experiment 718: Making Soap - Saponification

Section 1: Purpose and Summary

Make lye soap from sodium hydroxide and olive oil via the saponification reaction.

Soapmaking is an ancient technique which includes the mixing of water, a solution formed from burned wood ashes and fats and/or oils. The chemical reaction for soapmaking is called saponification. The modern technique to form solid soap uses plant oils and/or animal fat and sodium hydroxide to form solid bars or potassium hydroxide to form liquid soap. The saponification reaction breaks the oil or fat molecule into glycerin and a sodium or potassium salt of the long hydrocarbon chains that have a carboxylate end functional group.

There are many kinds of fats and oils, both animal and vegetable. Fats are usually solid at room temperature, but many oils are liquid at room temperature. The physical state of these fats and oils depends on the degree of unsaturation in the molecule. The more double bonds, the more likely the fat or oil will be a liquid at room temperature. Liquid cooking oils originate from corn, peanuts, olives, soybeans, and many other plants. For making soap, all different types of fats and oils can be used – anything from lard which comes from animal fat to exotic tropical plant oils like palm and coconut oils.

In this lab, you will test various fats and oils for their degree of saturation by reacting any double bonds in the fat or oil with bromine. Remember, that unsaturated carbon-carbon double and triple bonds can react with halogens to form halogenated hydrocarbons.

Addition Reaction Example:

Alkene or Alkyne + Br_2 → Bromoalkane

Saponification Reactions:

Fat + Lye → Soap + Glycerol

Additionally, you will explore water that has a high concentration of dissolved minerals which is called hard water. The minerals that can be part of hard water minerals can include calcium (Ca^{2+}), magnesium (Mg^{2+}), and/or iron(III) Fe^{3+} ions. These dissolved ions make it harder for soap to make a lather and be effective in solubilizing fats and dirt and removing them from your skin. These ions combine with the soap and precipitate into hard-water scum. You can also see dissolved minerals when they precipitate on your sink and bath/shower components as a white precipitate called scale. If your water comes from a water well, you may see this more often than if you get water from a municipal water source. Some people get around this problem, by using a water softener which removes the ions in the water by replacing them with sodium ions (Na^{+}).

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Be sure to exercise caution when dispensing the 9 M NaOH. If the chemical comes into contact with your skin, immediately rinse with water for a minimum of fifteen minutes and notify your instructor.

Personal Protective Equipment (PPE) required: safety goggles, lab coat, closed-toe shoes.

Waste Disposal:

After completing parts 1 and 2 of this experiment, rinse all glassware in the laboratory sink with soap solution and rinse completely with laboratory water.

After completing part 3, pour contents of test tubes into the organic waste container in the fume hood.

Section 3: Procedure

Part 1: Making Soap

Materials: warm olive oil (preheated by instructor in crockpot), 9 M sodium hydroxide solution, stearic acid

Equipment: tall 250 mL beaker, stirring plate and magnetic stir bar, plastic disposable pipets, silicone soap mold

1. Your instructor has a container of olive oil, preheated to 35°C, at a central location in a crock pot. Pour 100 ml of the warm oil into a tall 250 mL beaker. Add a magnetic stir bar to the beaker.
2. Add 30 ml of 9 M sodium hydroxide solution to the beaker carefully and slowly to the beaker. Don't let the sodium hydroxide splash on you or the counter.
3. Start the magnetic stirrer on low and don't let the solution splash out of the beaker. You must stir for 20-45 minutes. The mixture will slowly become smoother and more opaque; it should thicken to a pudding-like consistency. While you wait for this stirring to complete, you may do the **DETERGENT/SOAP AND HARD WATER and DEGREE OF SATURATION** portions of the lab.
4. Add approximately 1 teaspoon of stearic acid. This will serve as a hardener for the liquid soap. Stir on the magnetic stirrer for 5 more minutes. Remove the stir bar and return to the stockroom.
5. Pour into chosen mold shape. Label with your names and lab section number with masking tape on the silicone soap mold.
6. After pouring into the mold, the process will continue on its own. The soap will heat up and liquefy again, then cool off slowly, harden and dry. So, the soap must be left undisturbed for at least 12 hours. You will pick up your finished soap in lab next week.

Part 2: Soap and Detergent Effectiveness in Hard Water

Materials: A small amount of soap made from PART 1, commercial powdered detergent, pH paper, hard water solution, laboratory water

Equipment: Two 150 ml beakers, masking tape, glass stir rod, 3 test tubes, test tube rack

1. Label one beaker "soap" and one beaker "detergent". Put 100 ml of laboratory water in each beaker. Heat both beakers together on a hot plate.

2. Dissolve 2 g of the moist soap from Part 1 (use the leftover soap from your beaker in Part 1) in the boiling water of your beaker labeled “soap”. Dissolved 1 g of the commercial powdered detergent in the beaker labeled “detergent”. Use these two solutions to complete the data table.
3. Use pH paper and place the end of the paper into the solution in one of the beakers. Test both beakers and compare the pH of the soap solution compared to the detergent solution.
4. Place 4 drops of mineral oil into each of 3 test tubes. Add 5 ml of laboratory water into the first tube, add 5 ml of the soap solution into the second test tube, and add 5 ml of the detergent solution into the last test tube. Place your finger over the end of the test tube or place a cork in the end, shake the test tubes, then visually inspect the tubes. Record whether the solution emulsifies the oil. If the solution can surround and emulsify the oil, you won’t see a layer of oil at the top of the test tube. If the test tube looks the same top to bottom and side to side, then the oil, soap or detergent and water have formed a homogeneous mixture. This is what an emulsified mixture looks like. If the soap or detergent can’t surround the oil, you will see two layers: oil on top and water on the bottom.
5. Place 5 ml of soap solution into each of three test tubes. Add 2 ml of 0.5% $MgCl_2$ to the first tube, add 2 ml of 0.5% $CaCl_2$ to the second test tube, and add 2 ml of 0.5% $FeCl_3$ to the third test tube. Shake these three test tubes and look for a precipitate (white, chunky floating particles). Add 4 drops of mineral oil to each of the three test tubes. Shake them again and look for emulsification (like step 4 above).
6. Do the same process in three new test tubes with the detergent solution. Add the ionic solutions from Step 5 (above) to each of the three test tubes as you did for the soap solution. Check for precipitation. Add 4 drops of mineral oil to each of the three test tubes. Shake them again and look for emulsification (like Step 4 above). Record all your observations on the data sheet below.

	pH	**Observation**
Soap		
Detergent		
Laboratory water		

Soap and Mg^{2+} ion	**Soap and Ca^{2+} ion**
Observation	**Observation**
Precipitate (Y/N?)	**Precipitate (Y/N?)**

Emulsified (Y/N?)	**Emulsified (Y/N?)**

Detergent and Mg^{2+} ion	**Detergent and Ca^{2+} ion**
Observation	**Observation**
Precipitate (Y/N?)	**Precipitate (Y/N?)**
Emulsified (Y/N?)	**Emulsified (Y/N?)**

Part 3: Degree of Saturation of Various Fats and Oils

Materials: Olive oil, melted shortening, 2% bromine (Br_2) in dichloromethane, dichloromethane, pipet

Equipment: 2 test tubes, test tube rack

Add 2 drops of olive oil to 1 ml of dichloromethane in a test tube. Add 2% Br_2 in dichloromethane dropwise to the oil. Shake the test tube until the color disappears. Continue this process, and keep track of the number of drops, until you finally see the color stay in the solution. Do the process again with a fresh test tube and melted shortening. Compare how the two fats differ on the data table below.

Olive oil	**No. of drops of bromine used**
Shortening	**No. of drops of bromine used**

Which fat is more unsaturated?

Post Lab Questions:

1. Explain how soaps emulsify oils from your skin.

2. Draw an emulsified oil in a micelle.

3. How is a micelle similar to the bilayer of a cell wall? How is it different?

4. Soap doesn't kill bacteria, so how does soap work to clean your hands?

5. Draw the reaction of any alkene with Br2.

6. Explain how the bromine test works to detect unsaturation.

7. Which has more unsaturation: olive oil or shortening?

8. What does unsaturation have to do with the physical properties of an oil (physical state at room temperature)?

9. Which is more basic: your soap or detergent?

10. Which alkene below would be more unsaturated:

Alkene A- requires 10 drops of bromine in dichloromethane before you see color?

OR

Alkene B - requires 20 drops of bromine in dichloromethane before you see color?

Notes:

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1.26: Experiment_726_Paper Chromatography_1_2_1

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 726: Paper Chromatography**

Experiment 726: Paper Chromatography: A Technique of Separation and Identification

Section 1: Purpose and Summary

One of the problems encountered most frequently in chemistry is that of separating a mixture into the pure substances which compose it. Most natural materials, such as seawater, air, oil, coal, and so on are mixtures. In order to study these materials chemically, we must first treat the mixture in some way to separate it into single, pure substances. (A "pure substance" consists entirely of just one thing; meaning one kind of molecule.) Because it does consist of just one thing, it always behaves in the same way when tested. For example, pure water always boils at 100°C (212°F), provided that the test is done at atmospheric pressure.

Many different methods have been devised for separating mixtures into their components. In the present experiment, we will use a method called chromatography. It is used quite widely for making small-scale separations and identifications. The method works because of the differences in the ways various components of a dissolved mixture interact with a fixed solid. The fixed solid can be made of different materials and in different shapes, depending on the version of chromatography that is being done. One version is called paper chromatography.

To perform paper chromatography to separate components of a mixture, there are several steps. First, the mixture that is to be separated is dissolved (if it is not already in liquid form.) Then, a small drop of the solution is applied to a piece of special chromatography paper, which is a porous paper similar to filter paper. This drop makes a tiny spot on the paper. Many such spots (of different materials) may be placed side by side on the same piece of paper. Next, the spotted paper is made to stand in a small amount of some special solvent (liquid), in such a way that only the bottom edge of the paper is submerged in the liquid, not the spots. The paper acts like a wick, drawing the liquid up the paper by capillary action. The solvent then slowly rises up the paper, reaches the spots, and begins to dissolve them and carry the substances up the paper with it.

The key to this separation is that the different components of the mixture in each spot interact with the paper differently, and so will be found (after a few minutes) to have reached different heights on the paper. This happens because some components are more strongly attracted to the paper (and so move more slowly), while others are more strongly attracted to the solvent. Any particular substance always moves at the same rate, no matter what else it may have been mixed with originally. For that reason, it can always be recognized and identified, because it will always rise to the same height, relative to the heights that the other components rise.

The movement of any spot on the paper can be quantified by calculating its R_f (*retention factor*) value after you have stopped the experiment and the paper has dried.

R_f = distance traveled by the solute

distance traveled by the solvent front

The distances used in calculating R_f values are measured as shown in the following figure. To determine the *distance traveled by the solute*, measure from the point at which you originally applied the spot to the center or densest part of the spot. The *distance traveled by the solvent front* is measured from the original point of application of the spot to the limit of movement of the solvent front (which must be marked immediately after the paper is removed from the beaker, because it may be nearly invisible after the solvent evaporates).

If all conditions could be maintained constant, R_f values would be constant. However, either variations in temperature or in the

composition of the solvent phase or changes in the paper can alter the R_f value. The R_f value is useful mainly for expressing the relative mobility of two or more solutes in a particular chromatographic system. The absolute R_f values may change from day to day, but their values in relation to each other remain nearly constant.

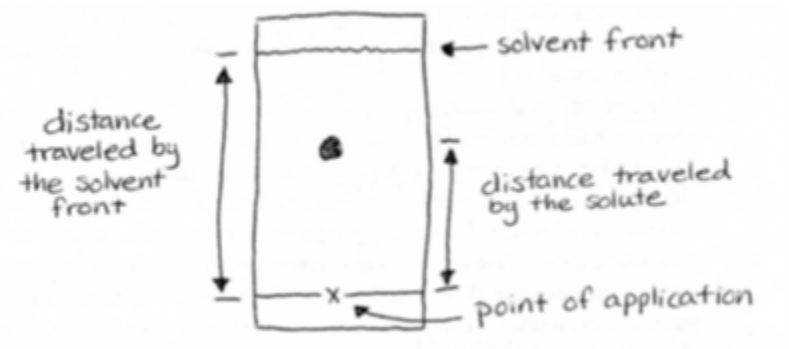


Figure 1: Completed paper chromatography containing only 1 dye.

In this experiment, students will measure the values of several dyes in 3 different solvent systems. Students will also analyze an unknown mixture of dyes in order to identify the dyes present in the mixture. The three different solvent systems are 1) laboratory water, 2) an aqueous solution of 0.10% sodium chloride (table salt), and 3) rubbing alcohol (70% isopropyl alcohol and 30% water). By using different solvents, the dyes will travel differently in paper chromatography, and the R_f values will be different. The goal is to find a solvent that gives a different value for every dye as this will separate all the dyes from each other (scientists say that the dyes are ***resolved*** when the solvent system creates distinctly different values for each dye.) When every dye is separated from all the other dyes, then you can match up R_f values and identify each dye in the unknown mixture.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

The rubbing alcohol used in this lab gives off fumes, so keep the beaker containing this solvent covered with plastic wrap when it is not in the hood.

Waste Disposal:

The rubbing alcohol should be disposed of in the organic waste container in the hood. The aqueous solvents may be poured down the drain. The chromatography paper can be disposed in the trash.

Section 3: Procedure

Preparing and developing the chromatogram

1. Obtain three (3) rectangular pieces of chromatography paper. Touch them only at the edges. (Oils from your hands can affect the separation.) All 3 pieces will be set up the same way: the only difference will be that a different solvent mixture will be used for each.	
2. Make sure that the chromatography paper will fit into the beaker you plan to use when it is rolled into a cylinder. Make sure that the paper will not touch the sides of the beaker.	
3. **Use a pencil** (not a pen) to make a straight line all the way across the long way of the paper, at a distance of about one centimeter above the bottom edge (in Figure 1, this line has an “X” in it).	

4. **You will be testing several dyes as well as your unknown mixture: each chromatography paper will have all the same component dyes including the unknown mixture**. For each dye that you test, make a small, light circle in pencil on the line, spacing the circles evenly apart on the paper. Next to each circle put an identifying letter or code so that you will know what the spot contained. (These circles are the "point of application" for each dye: see Figure 1.)	**Which Unknown Mixture did you use?** **Unknown Mixture #_____**
(Use this space for drawing a sketch of your chromatography paper. Just sketch 1 chromatography paper because all 3 should be exactly the same. In the same below also recording the identifying letters in the same order they appear on the rectangular piece of chromatography paper. Include enough information here so you know what each identifying letter stands for.)	**List your identifying letters in the same order they appear on the rectangular piece of chromatography paper:**
5. Apply tiny drops of each liquid to be tested to the appropriate spot on the pencil line. This can be done by repeatedly dipping a toothpick into the liquid and then touching it to the paper. **The darker and smaller your spots are, the better results you will obtain**. You may wish to practice one spot first on a scrap of paper towel before spotting your chromatography paper. The chromatography paper is expensive, so please do not waste it.	
6. Roll the spotted paper into a cylinder with the spots on the outside and staple the ends together. Use two staples, placing them about one-third of the way from the upper and lower edges. **Staple the paper so that the ends of the paper do not touch each other**, because if they do, it will affect the flow of the solvent at that point.	
7. Obtain 3 clean, dry beakers. A 250-mL beaker is a good size for this experiment, but any size can work if it has enough room for the rolled up chromatography paper without touching the sides of the beaker. Label each beaker with a different solvent system. The 3 different solvent systems are 1) laboratory water, 2) an aqueous solution of 0.10% sodium chloride (table salt), and 3) rubbing alcohol (70% isopropyl alcohol and 30% water).	
8. To the empty beaker, add a few milliliters of the appropriate solvent to each beaker. Use just enough solvent so that it will wet the bottom edge of the cylinder of paper but not deep enough to touch the spots.	
9. Write the name of the solvent you are using at the top of the chromatography paper, and place it in each beaker. Carefully stand the paper cylinder in the beaker that has the solvent in it. Make sure that the paper does not touch the sides of the beaker. Remember that the spots must not be immersed in the solvent. Cover the beaker with plastic wrap or aluminum foil.	
10. Let the beaker stand undisturbed. Do not pick it up or slide it around because that will cause sloshing. Watch the solvent rise up the paper (it looks wet where solvent is), and watch the original spots spreading out into different colored components.	

11. The chromatogram is finished when the separations are complete. However, since separations are not always perfect, **you should stop yours when the solvent has risen about three-fourths of the way** toward the top of the paper, even if the results are not absolutely perfect. (Sometimes it is necessary, in actual scientific research, to try a different solvent or use a larger piece of paper.)	
12. Stop the chromatography by lifting the paper out of the beaker. Immediately mark (with a pencil) the height reached by the solvent. (This is called the solvent front and is useful as a reference point.) Unless otherwise specified by local requirements, all solutions used in this experiment may be disposed of down the drain.	
13. Let the paper dry. You may use a hair dryer if you wish to speed up the process.	

Section 4: Analysis of the Chromatograms

For each spot, measure the distance (in centimeters) traveled by the spot (use the center of the spot) and the distance traveled by the solvent front. Record the color of each spot, then calculate *Rf* values for each of the spots.	**Unknown Mixtures** may contain 2 or 3 or 4 different component dyes mixed together. Measure every component dye in your unknown mixture.

Chromatograph using **laboratory water** Distance the **solvent front** moved = _____ cm

Name of dye	Color of dye	Distance dye moved	R_f values

Unknown Mixture:	Color of dye	Distance dye moved	R_f value
Component dye #1			
Component dye #2			
Component dye #3			
Component dye #4			

Chromatograph using **0.10% salt water** Distance the **solvent front** moved = _____ cm

Name of dye	Color of dye	Distance dye moved	R_f values
Unknown Mixture:	Color of dye	Distance dye moved	R_f value
Component dye #1			
Component dye #2			
Component dye #3			
Component dye #4			

Chromatograph using **rubbing alcohol** Distance the **solvent front** moved = _____ cm			
Name of dye	Color of dye	Distance dye moved	R_f values
Unknown Mixture:	Color of dye	Distance dye moved	R_f value
Component dye #1			
Component dye #2			
Component dye #3			
Component dye #4			

Section 5: Identifying the components in the Unknown Mixture

Use the colors and the R_f values of the known dyes to identify the dyes found in your Unknown mixture.	**Unknown Mixture # _____**
Component dye #1 =	
Component dye #2 =	
Component dye #3 =	
Component dye #4 =	

Post Lab Questions:

1. Why use a pencil and not a pen to mark your chromatograms?

2) Why is it important that the chromatography paper not touch the sides of the beaker?

3) Which solvent system worked the best for you? Explain.

4) Suppose a student did today's experiment and obtained the following results:

Solvent system	Dye #1	Dye #2	Dye #3
Laboratory water	R_f = 0.64	R_f = 0.38	R_f = 0.55
0.10% salt water	R_f = 0.92	R_f = 0.27	R_f = 0.59
Rubbing alcohol	R_f = 0.79	R_f = 0.43	R_f = 0.45

4a) Which solvent is best for separating Dyes #1-3?

4b) Assume that the student receives an Unknown mixture that contains Component Dye #2 and Component Dye #3. Draw the chromatogram the student would expect to obtain when analyzing Dye #1, Dye #2, Dye #3 and her "Component" mixture when using the best solvent, the same solvent you picked in question 4a. Include the points of application and the solvent front in your drawing.

Notes:

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1.27: Experiment_727_Organic Compound Functional Groups__1_2_0

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 727: Organic Compound Functional Groups**

Experiment 727: Organic Compound Functional Groups

Section 1: Purpose and Summary

- Determine the solubility of different organic compounds.
- Determine the reactivity of functional groups with various test reagents.
- Identify an unknown organic compound based on its functional group.

The functional group in an organic compound is the part of the molecule that undergoes a structural change during a chemical reaction. It also influences the solubility of the compound in water or in organic solvents. Certain chemical tests will allow the identification of a specific functional group in a compound. The functional groups that will be analyzed in this experiment are alcohols, phenols, aldehydes, ketones, carboxylic acids, and amines.

In this experiment, students will determine the solubility of different organic compounds in water, dilute acid, or dilute base. This solubility can provide information on what functional group is present or whether the compound has a high or low formula mass. Students will perform chemical tests for known organic compounds to suggest the presence or determine the absence of a specific functional group in the compound. Students will use the results of both solubility and functional group tests to generate experimental data that will help them identify an unknown sample.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

Wash your hands after working with organic reagents and solutions. Do not eat in the laboratory.

Waste Disposal:

While you are doing the experiment, collect all waste into a beaker.

When you are finished with the experiment, pour the contents of the waste beaker into the organic waste container in the fume hood.

Section 3: Procedure

Important: Use clean, dry and labeled small or medium-size test tube for each test.

Known compounds include: *2-butanone, benzaldehyde, n-butyl bromide, cyclohexene, 2-propanol, tert-butyl alcohol, isobutyl alcohol, propanoic acid, triethyl amine, phenol.*

Part 1: Solubility Tests

Low molecular weight hydrocarbons are soluble in water, while high molecular weight ones are not. Carboxylic acids are soluble

in basic solutions, while amines are soluble in acidic solutions.

A. Solubility in water

1. Into a small test tube, place 5 drops of known sample. If the compound is in solid form, add a pea-sized sample into the test tube.
2. To each sample, add 10 drops of laboratory water and mix thoroughly by holding the top of the test tube and flicking the bottom with your finger. Does the sample dissolve in water? (Note: A liquid sample that is insoluble in water forms a second liquid layer.) Record your observations.
3. If the compound is soluble in water, test its acidity or basicity using litmus paper. If blue litmus turns red, it is acidic. If red litmus turns blue, it is basic. Record your observations.

B. Solubility in 5% NaOH

1. Into a small test tube, place 5 drops of known sample. If the compound is in solid form, add a pea-sized sample into the test tube.
2. To each sample, add 10 drops of 5% NaOH. Mix thoroughly by holding the top of the test tube and flicking the bottom with your finger. Record your observations.

C. Solubility in 5% HCl

1. Into a small test tube, place 5 drops of known sample. If the compound is in solid form, add a pea-sized sample into the test tube.
2. To each sample, add 10 drops of 5% HCl. Mix thoroughly by holding the top of the test tube and flicking the bottom with your finger. Record your observations.

Part 2. Functional Group Tests

A. Silver Nitrate Test for Alkyl Halides and Carboxylic Acids

n-butyl bromide, propanoic acid

1. Into a clean, dry small test tube, place 20 drops of 0.1 M solution of $AgNO_3$ in 95% ethanol.
2. Add **one drop** of known sample and mix thoroughly. Observe for the formation of white or yellow precipitate.
3. If no reaction is observed within five minutes at room temperature, warm the mixture in a beaker of boiling water (on a hot plate and add boiling chips to the bath), and observe any change. Record your observations.

4. If precipitate forms, add several drops of 1 M HNO_3 and note any changes, particularly dissolution of precipitate. Record your observations.

B. Chromic Acid Test for Alcohols and Aldehydes

2-propanol, tert-butyl alcohol, isobutyl alcohol, benzaldehyde

1. Into a small test tube, place 20 drops of known sample.
2. Add **one drop** of the chromic acid reagent. Mix thoroughly by holding the top of the test tube and flicking the bottom with your finger. Note the color change of the solution. A positive test gives a change in color of the reagent from orange to green. A blue color may also be observed and is a positive test. Record your observations.

C. Bromine Test for Alkanes and Alkenes

cyclohexene

1. Into a small test tube, add 5 drops of known sample.
2. To each sample, add 1 drop of 2% solution of bromine in dichloromethane. Mix thoroughly by holding the top of the test tube and flicking the bottom with your finger. Note the time required for the decolorization of the bromine solution. Record your observations.

D. Ferric chloride Test for Phenols

Phenol

1. Into a small test tube, mix 2 – 3 drops of known sample and 2 drops of laboratory water.
2. Add 1 – 2 drops of 2.5% $FeCl_3$. Stir the mixture well with a stirring rod. Observe immediately and note the color change of the solution. A positive test is indicated by the change in color of the $FeCl_3$ solution from yellow to a blue green, violet, grey, or red.

E. Iodoform Test for Ketones (carbonyl on C-2) or 2^o Alcohols (OH on C-2)

2-propanol, 2-butanone

1. Into a small test tube, add 2 – 3 drops of known sample and 1 mL of laboratory water. Mix thoroughly by swirling.
2. Add 1 mL of 10% NaOH, followed by 1.5 mL of 0.1 M I_2/KI solution. Note the disappearance of the brown color of the I_2/KI solution followed by the formation of a light-yellow precipitate. Record your observations.

F. 2,4-DNP Test for Aldehydes and Ketones

benzaldehyde, 2-butanone

1. Into a small test tube, place 20 drops of 2,4-DNP solution.
2. Add 2 drops of the known sample and mix thoroughly by swirling. Observe for the formation of an orange or red-orange precipitate. Record your observations.

G. TCICA Test for Alcohols

2-propanol, tert-butyl alcohol, isobutyl alcohol

1. Into a small test tube, add 10 drops of the TCICA solution in acetonitrile (30 mg/mL), one drop of 1 M HCl, and one drop of the known sample.
2. Mix thoroughly by holding the top of the test tube and flicking the bottom with your finger. Note the time required for the precipitate to form. Record the elapsed time.

Part 3: Unknown Compound

1. Obtain a sample of an unknown compound from your instructor. **Record the identification code of the unknown.** This is very important! With no identification, your work cannot be graded.
2. Perform solubility and functional group tests and tabulate your results.
3. Identify the functional group of the unknown sample based on your results.

Section 4: Data and Results

KNOWN Compound Solubility and Functional Group Tests

Part 1: Solubility Tests

Known Compounds	**Observations**		
	Water/Litmus	**5% NaOH**	
n-butyl bromide			
isobutyl alcohol			

2-propanol			
tert-butyl alcohol			
phenol			
benzaldehyde			
2-butanone			
propanoic acid			
triethylamine			
cyclohexene			

Part 2: Functional Group Tests

1. Silver Nitrate Test

Compounds Tested	**Observations**
n-butyl bromide	
propanoic acid	

2. Chromic Acid Test

Compounds Tested	**Observations**
isobutyl alcohol	
2-propanol	
tert-butyl alcohol	

benzaldehyde	

3. Bromine Test

Compound Tested	**Observations**
cyclohexene	

4. Lucas Test

Compounds Tested	**Observations**
isobutyl alcohol	
2-propanol	
tert-butyl alcohol	

5. Ferric Chloride Test

Compound Tested	**Observations**
phenol	

6. Iodoform Test

Compound(s) Tested	**Observations**
2-propanol	
2-butanone	

7. 2,4-DNP Test

Compound(s) Tested	**Observations**

benzaldehyde	
2-butanone	

8. TCICA Test

Compound(s) Tested	**Observations**
isobutyl alcohol	
2-propanol	
tert-butyl alcohol	

UNKNOWN Compound Solubility and Functional Group Tests

Identification Code for the Unknown: _____

Fill the following table, where applicable.

Chemical Test	**Observations**	**Conclusi**
Water solubility/Litmus		
5% NaOH solubility		
5% HCl solubility		
Silver Nitrate Test		
Chromic Acid Test		
Bromine Test		
Ferric Chloride Test		

Iodoform Test		
2,4-DNP Test		
TCICA Test		

Functional group present in unknown: _____

High or low molecular weight? _____

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1.28: Experiment_728_Qualitative Testing of Carbohydrates 1_1

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 728: Qualitative Analysis of Carbohydrates**

Experiment 728: Qualitative Analysis of Carbohydrates

Section 1: Purpose and Summary:

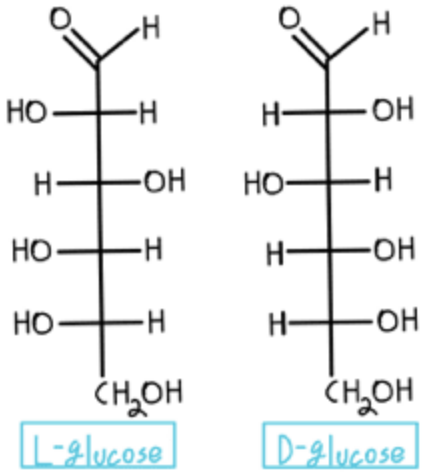
- Develop an understanding of what carbohydrates are.
- Identify different types of carbohydrates.
- Observe how different carbohydrates react in different chemical tests.

What are carbohydrates?

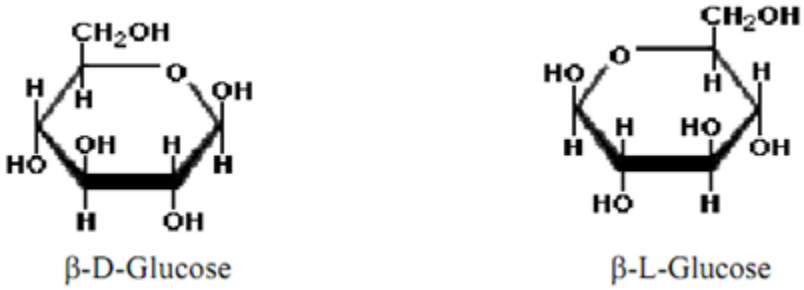
Carbohydrates are a class of natural compounds that contain either an aldehyde or a ketone group and many hydroxyl groups – they are often called polyhydroxy aldehydes or ketones. A *monosaccharide* consists of a single carbohydrate molecule, containing between 3 and 7 carbons. Glucose and fructose are examples of monosaccharides. A *disaccharide* consists of two monosaccharides that are linked together. Sucrose and lactose are disaccharides. A *polysaccharide* consists of many monosaccharides linked together. Starch, pectin, glycogen, and cellulose are examples of polysaccharides.

Carbohydrates are used for energy. The carbohydrates that we eat are broken down in our bodies and eventually form water and carbon dioxide. The energy obtained in this process is used for other reactions that must occur in the body. Excess carbohydrates that we eat can be stored in the liver as glycogen or can be converted to fats. Plants create carbohydrates in the process of photosynthesis, where energy from the sun is used to build carbohydrates from water and carbon dioxide.

Monosaccharide structures can be written as Fischer projections (they are named after Emil Fisher who first used them in 1891). In a Fischer projection, the structure is drawn vertically with the carbonyl carbon at the top. It is understood that for each chiral carbon in the molecule, the horizontal bonds point out of the page (toward you) and the vertical bonds point into the page (away from you). Fischer projections are used to indicate the stereochemistry of each chiral carbon in the molecule and to compare monosaccharide structures easily. For example, there are many compounds with the same connections of atoms but different stereochemistry, and they all have different names! The Fischer projections for glucose and galactose are shown below. Note that the only difference between these sugars is the stereochemistry around carbon 4, yet they have different names.



In solution, most monosaccharides exist in a cyclic form – the aldehyde or ketone group reacts with one of the –OH groups on the other end of the same molecule to form a cyclic hemiacetal. Shown here are the cyclic structures for D-glucose. Notice that there are two possibilities: α-D-glucose and β-D-glucose. These are called the different anomers of glucose. In solution, there is an equilibrium between the cyclic form and the open chain or free aldehyde form. The rings are constantly opening up and closing again. In this way, the alpha and beta forms can be interconverted.



Chemical Tests for Carbohydrates

A *reducing sugar* is one that can be oxidized. In order to be a reducing sugar, the molecule must contain a free anomeric carbon, since it is the open-chain form of the aldehyde that is able to react (and be oxidized). One test for reducing sugars involves **Fehling's reagent**, which contains Cu^{2+} ions in an aqueous basic solution. If a reducing agent is present, the Cu^{2+} is reduced to Cu^{+} and forms a red precipitate of Cu_2O. Therefore, if Fehling's solution is added to a solution containing a reducing sugar, a red precipitate will form. Sometimes the reaction mixture must be heated in order for the precipitate to form. The color of the precipitate can vary from red to orange to green (the green color is actually a mixture of an orange and a blue precipitate).

Barfoed's test is similar to Fehling's test, except that in Barfoed's test, different types of sugars react at different rates. Barfoed's reagent is much milder than Fehling's reagent. Reducing monosaccharides react quickly with Barfoed's reagent, but reducing disaccharides react very slowly or not at all. Therefore, it is possible to distinguish between a reducing monosaccharide and a reducing disaccharide using Barfoed's reagent. A positive test is a dark red precipitate and is evidence of a reducing monosaccharide.

In **Seliwanoff's test**, a dehydration reaction is involved. Seliwanoff's reagent contains a non-oxidizing acid (HCl) and resorcinol. When a ketose (sugars with a ketone group) is reacted with this reagent, it becomes dehydrated and a cherry-red complex forms (not a precipitate). Aldoses (sugars with an aldehyde group) also react with this reagent, but much more slowly than ketoses. When Seliwanoff's reagent is reacted with a disaccharide or a polysaccharide, the acid in the solution will first hydrolyze them into monosaccharides, and the resulting monosaccharides can then be dehydrated. Disaccharides and polysaccharides will therefore react slowly with Seliwanoff's reagent. When you carry out this test, it is important to note the time required for a reaction to

occur.

Iodine forms a blue, black, or gray complex with starch and is used as an experimental test for the presence of starch. The color of the complex formed depends on the structure of the polysaccharide and the strength and age of the iodine solution. Iodine does not form a complex with simpler carbohydrates (monosaccharides and disaccharides). Amylose (starch) is helically coiled in solution, and it is this helical structure that is necessary to form the blue complex with iodine. Monosaccharides and disaccharides are too small to be helically coiled. Amylopectin, cellulose, and glycogen

form different colors with iodine – red, brown, or purple.

Many carbohydrates can undergo **fermentation** in the presence of yeast. The carbohydrate is the food source for the yeast, and the products of the fermentation reaction are ethanol and carbon dioxide gas.

$C_6H_{12}O_6 \rightarrow 2\ CH_3CH_2OH + 2\ CO_2\ (g)$

Glucose Ethanol

Fermentation is used in the processes of making beer and wine, where the alcohol produced by the yeast is the desired product. Not all sugars, however, can be used by yeast as a food source. You will test which sugars ferment in the presence of yeast and which ones do not. The evidence of fermentation will be the evolution of carbon dioxide gas. In the test, a quantity of a solution (containing yeast and the sugar to be tested) will be trapped in an upside-down small test tube. After a few days, you will check to see if a gas bubble has formed in the test tube. If there is a gas bubble, it means fermentation did occur.

Disaccharides and polysaccharides can be **hydrolyzed** in the presence of acid or specific enzymes. When a disaccharide is hydrolyzed, the products are the individual monosaccharides. When a polysaccharide is hydrolyzed, the products will depend on how long the mixture is allowed to react, the concentration of acid or enzyme, and other factors. Polysaccharides are very long and have many glycosidic bonds to hydrolyze. They cannot all be hydrolyzed at the same time, so the product is a mixture of dextrin, maltose, and glucose. If a polysaccharide sample is hydrolyzed completely (which means that it must react for a while), the product is glucose. In this experiment, you will hydrolyze a sample of sucrose and then test it for the presence of a reducing sugar. You will also hydrolyze a sample of starch and then test it for the presence of both a reducing sugar and starch.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Wear your safety goggles.

Waste Disposal:

At the end of the experiment, all wastes go into the inorganic waste container.

Section 3: Procedure

Note: Several tests require a hot water bath. Start several beakers with water heating on a hot plate to have them available when you need them. **1.Fermentation** In this part of the experiment, an instructor will have set up a test for glucose, fructose, lactose, sucrose, starch, water. This part describes how the test has been prepared. Large test tubes have been labeled and filled each one with the solution to be tested. A small test tube was placed upside-down in each large test tube. The top of each large test tube was covered, and inverted so that the small test tube inside gets filled completely with the solution. The small test tube starts as completely filled with solution – it does not have any gas bubbles in it. To each test tube, 0.5 g of the sample of the carbohydrate, 50 ml laboratory water and 0.02-0.03 g of yeast was added and dissolved. Note the date and time that the demonstration was started. Check to see if there is a gas bubble in any of the small test tubes. The presence of a gas bubble is evidence that a gas was produced in the reaction. If a gas was produced, that means that fermentation occurred in the tube. Record your observations.	Observations: _____ _____ _____ _____ _____ _____ _____ _____ _____ _____
2. Fehling's Test In this part of the experiment, you will test known samples of glucose, fructose, lactose, sucrose, starch, and compare with a sample of a solution with an unknown component. Add 6 drops of each solution to be tested to each of 6 labeled test tubes. Fehling's solution is not stable and must be made prior to use by combining two solutions, called A and B. In a larger test tube, mix 6 mL of Fehling's solution A with 6 mL of Fehling's solution B. Add 2 mL of this combined Fehling's solution to each of the 6 labeled test tubes, and mix each tube thoroughly by shaking the tube well. Place these tubes in a boiling water bath for 5 minutes. After 5 minutes, remove the tubes from the water bath and record your observations. The formation of a red precipitate indicates a positive reaction.	Observations: _____ _____ _____ _____ _____ _____ _____

3. Barfoed's Test In this part of the experiment, you will again test known samples of glucose, fructose, lactose, sucrose, starch, and compare with a sample of a solution with an unknown component. Add 1 mL of each solution to be tested to each of 6 labeled test tubes. Add 3 mL of Barfoed's reagent to each of the 6 test tubes, and mix each tube thoroughly by shaking the tube. Place these tubes in a boiling water bath for 5 minutes. After 5 minutes, remove the tubes from the water bath, let them cool, and then cool them further by running cold water over the outside of each test tube. Record your observations. The formation of a red precipitate indicates a positive reaction. Note the amount of time needed for the red precipitate to occur in each case.	Observations: _____ _____ _____ _____ _____ _____ _____ _____
4. Seliwanoff's Test For this part, you will test glucose, fructose, lactose, water, and compare with a sample of a solution with an unknown component. Add 10 drops of each solution to be tested to each of 5 labeled test tubes. Add 4 mL of Seliwanoff's reagent to each of the 5 test tubes, and mix each tube thoroughly by shaking the tube. Place these tubes in a boiling water bath and note the time needed for any color change to occur. After 10 minutes, stop heating the tubes. Record your observations. A color change indicates a positive reaction.	Observations: _____ _____ _____ _____ _____ _____
5. Iodine Test Test glucose, fructose, lactose, sucrose, starch, water, and compare with a sample of a solution with an unknown component. Add 1 mL of each solution to be tested to each of 7 labeled test tubes. Add 3 drops of iodine solution to each of the 7 test tubes, and mix each tube. Compare the colors and record your observations.	Observations: _____ _____ _____ _____

6. Hydrolysis This procedure is separated into 3 parts: **6A.** Add 0.5 mL of 3 M HCl to 5 mL of a 1 % sucrose solution in a test tube. Mix. Heat and stir the mixture in a boiling water bath for 20 minutes. (You may add deionized water to this solution if the volume starts getting low.) Cool the solution, and add 1 M NaOH until the solution tests neutral on pH paper. Transfer 8-10 drops of this solution to a small test tube. In a separate tube, mix together 1 mL of Fehling's solution A with 1 mL of Fehling's solution B. Add this mixture to the small test tube containing your hydrolyzed sucrose, and heat for a few minutes in a boiling water bath. Record your observations. Compare the results of this test with your results for sucrose that has not been hydrolyzed in part 2 of this experiment.	Observations: _____ _____ _____ _____ _____ _____ _____
6B. Place 3 mL of 1 % starch in a test tube and add 0.5 mL of 3 M HCl. Mix and place this mixture in a boiling water bath for 10 minutes. After 10 minutes, remove the tube from the water bath and let it cool. Neutralize this solution with 1 M NaOH and mix well (use the same procedure for neutralization that you used in step 6A). Transfer 8-10 drops of this solution to a small test tube. (Save the rest of it for step 11.) In a separate tube, mix together 1 mL of Fehling's solution A with 1 mL of Fehling's solution B. Add this mixture to the small test tube containing your hydrolyzed starch, and heat for a few minutes in a boiling water bath. Record your observations. Compare the results of this test with your results for starch that has not been hydrolyzed in Part 2 of this experiment.	Observations: _____ _____ _____ _____ _____ _____ _____
6C. Using your solution from the end of step 6B (the hydrolyzed starch solution), transfer 1 mL to a small test tube. Add 3 drops of the iodine solution, and record your observations. Compare your results for this test with you results for starch that has not been hydrolyzed in Part 5 of this experiment.	Observations: _____ _____ _____

Post Lab Questions:

1. According to the results of each part of the experiment, identify your unknown and explain your reasoning.

2. Compare the results you obtained for the Fehling's test of sucrose to the Fehling's test of hydrolyzed sucrose. Explain your results.

3. Compare the results you obtained for the Fehling's test of starch to the Fehling's test of hydrolyzed starch. Explain your results.

4. Compare the results you obtained for the iodine test of starch to the iodine test of hydrolyzed starch. Explain your results.

5. What is meant by the term "reducing sugar"?

6. What is the purpose of testing water in the Seliwanoff's test and the iodine test?

7. Draw the ring structures for α-D-fructose and for β-D-fructose.

8. An unknown carbohydrate gave a red precipitate when tested with Fehling's reagent, turned red when reacted with Seliwanoff's reagent, and quickly gave a red precipitate when reacted with Barfoed's reagent. What conclusions can be made about this carbohydrate?

9. What test could be used to differentiate between sucrose and lactose? Explain.

10. What test could be used to differentiate between glucose and starch? Explain.

11. What test could be used to differentiate between glucose and fructose? Explain.

12. Why don't all of the disaccharides undergo fermentation with yeast?

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1.29: Experiment_729_Qualitative Testing of Amino Acids and Proteins 1_2

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 729: Qualitative Analysis of Amino Acids and Proteins**

Experiment 729: Qualitative Analysis of Amino Acids and Proteins

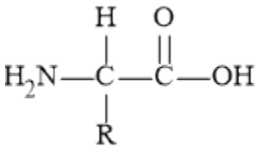
Section 1: Purpose and Summary:

- Develop an understanding of the structure and bonding in amino acids and proteins.
- Identify different amino acids and an unknown by paper chromatography.
- Observe how different amino acids and proteins react in different chemical tests.

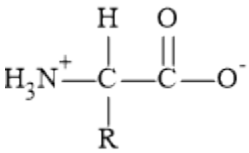
What are proteins?

Proteins are polymers of amino acids linked together by peptide bonds. Proteins are important biological molecules with many functions. There are transport proteins (hemoglobin is an example), storage proteins, structural proteins, proteins for muscular contraction, and others. Proteins that catalyze biological reactions are called **enzymes**. All proteins are very large molecules with high molecular weights that range from 6500 to 205,000 or more grams per mole.

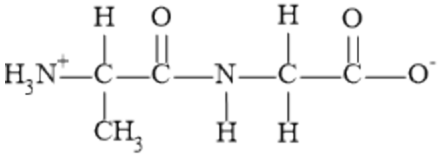
An **amino acid** is a molecule that contains an amino group and a carboxyl group in the same molecule. Amino acids found in proteins are α-amino acids. This means the amino group (NH_2- or NH_3^+-) is attached to the alpha carbon--this is the carbon next to the carboxyl group. There are 20 amino acids that differ from each other only in the identity of the side chain attached to the alpha-carbon. The amino acid side chains can be classified based on whether they are nonpolar, polar, acidic, or basic. The general structure of an amino acid is shown below. In the following structure, R represents the side chain.



Since proteins are biological molecules, they are usually found in a neutral solution that is buffered between pH 7.0 and 7.4. In this situation, the acidic and basic groups on the amino acids will be ionized. Most amino acids exist as "**zwitterions**" (dipolar ions) at pH 7. The structure of a zwitterion is shown below. Because of the charges, amino acids are water-soluble.



Amino acids can be linked together – the amino group on one amino acid can react with the carboxyl group on another amino acid, forming an amide. Therefore, the bonds linking together the amino acid residues are amide bonds, but in proteins they are called **peptide bonds**. Shown on the next page is the structure of a **dipeptide** (the prefix "di" means two--there are two amino acids in a dipeptide). A typical protein molecule contains around 100 amino acids joined by peptide bonds.



alanylglycine, a dipeptide

There are several aspects to describe structure of a protein. The **primary structure** of a protein is the sequence of amino acids. This sequence determines the overall shape of the protein. The **secondary structure** consists of regular and repeating structures held together by hydrogen bonds between the C=O and the N-H groups along the backbone of the molecule. The alpha helix and beta sheet are examples of secondary structure. The **tertiary structure** refers to the overall folding of the entire polypeptide chain and is determined by interactions between the side chains off the amino acids. These interactions include hydrogen bonding, hydrophobic interactions, salt bridges, and disulfide bridges. **Quaternary structure** involves the same types of interactions as tertiary structure, but the interactions occur between the side chains on different polypeptide chains. These interactions hold together different subunits of a protein.

If the overall folding of a protein is changed, the protein is called **denatured**. A denatured protein is no longer biological active. Some common denaturing agents include heat, organic solvents, acids, bases, agitation, detergents, and heavy metal ions. It is important to note that denaturation affects the secondary, tertiary, and quaternary structure of a protein, but does not affect the primary structure. Sometimes the denaturation is *reversible,* and the protein can be renatured (which reestablishes its biological activity).

Paper Chromatography of Amino Acids

Chromatography is a technique of separation and identification. There are many types of chromatography, including but not limited to paper chromatography, thin layer chromatography, gas chromatography, liquid chromatography, and ion-exchange chromatography. In this experiment, paper chromatography will be used to identify amino acids.

For paper chromatography, the samples to be tested are spotted on one edge of a rectangular piece of filter paper. The paper is placed in a container that has a solvent that wets the bottom of the paper. The paper acts as a wick for the solvent. The solvent passed through the samples as it wicks up the paper, carrying the samples. The samples move at different rates: those that are more attracted to the solvent will travel faster, and those that are more attracted to the paper will move slower. After the solvent has moved about 3/4 of the way up the paper, the chromatography is complete. The paper is removed from the container and dried. The amino acid solutions are colorless, so a developer needs to be used to make the amino acids visible. For amino acids, it is sprayed with a developer called ninhydrin. This will react with amino acids and produce a purple color. Ninhydrin will also react with proteins.

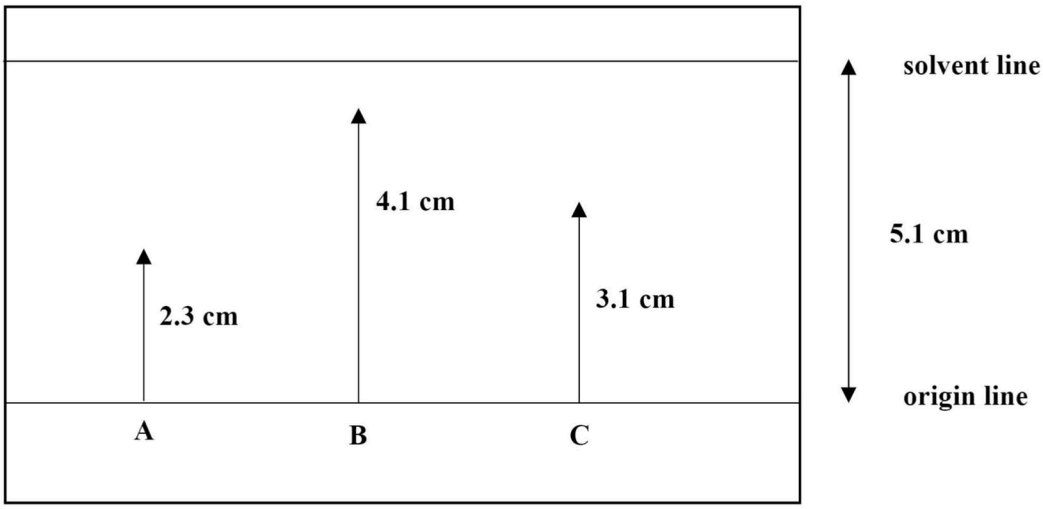
A photo of the chromatography setup is below:

<< PHOTO WILL BE IN NEXT UPDATE >>

After the paper is developed, the distance that the solvent traveled and the distance that each amino acid has traveled are measured. This is called the retention factor, R*f*, and has a different value for each amino acid is then calculated. By comparing R*f* values of unknown amino acids to those of known values, the unknowns can be identified.

A sample calculation of R*f* values is shown below.

$$R_f = \frac{\text{distance traveled by amino acid}}{\text{distance traveled by solvent}}$$



Spot A: $R_f = \frac{2.3\text{ cm}}{5.1\text{ cm}} = 0.45$ **Spot B:** $R_f = \frac{4.1\text{ cm}}{5.1\text{ cm}} = 0.80$

Spot C: $R_f = \frac{3.1\text{ cm}}{5.1\text{ cm}} = 0.61$

In this experiment, students will test several known amino acid solutions and one unknown. The results for the unknown should match one of the known amino acids.

Reactions of Amino Acids and Proteins

Hydrolysis

Proteins can be hydrolyzed either partially or completely in the presence of acids, bases, or digestive enzymes. When a protein is hydrolyzed, some or all of the peptide bonds are broken. The products obtained depend on how long the hydrolysis is allowed to take place. The products of partial hydrolysis are peptides and the products of complete hydrolysis are amino acids.

Biuret Test

Compounds that contain two or more peptide bonds will react with Cu^{2+} in a basic solution to form a violet-pink complex. The original Cu^{2+} solution is blue, so if the solution remains blue, the compound being tested could be an amino acid or a dipeptide or neither.

Xanthoproteic Test

The aromatic rings on tyrosine and tryptophan react with nitric acid. In this reaction, the aromatic rings become nitrated. When nitric acid is added to a sample and the mixture is heated, a yellow solution will result if the sample contains tyrosine or tryptophan. When this yellow solution is treated with a strong base (such as NaOH), it turns orange. Since most proteins contain one or both amino acids, most proteins will show a positive reaction in this test.

Ninhydrin Test

Free amino groups will react with the ninhydrin reagent to yield a purple solution. Almost all amino acids contain a free amino group (except proline and hydroxyproline). Some proteins also give a positive test with ninhydrin.

Sulfur Test

Sulfur-containing amino acids include cysteine and methionine. If a sample that contains one or both of these amino acids is acidified, the gas H_2S is produced, which smells like rotten eggs. When a piece of moistened lead (II) acetate paper is held over the solution as the H_2S is being produced, the H_2S reacts with the lead ion, forming a black or gray coating of PbS (lead (II) sulfide). Appearance of this black color is taken as a positive test for a sulfur-containing amino acid.

Denaturation

There are many ways to denature a protein. Recall that denaturation refers to a disruption of the secondary, tertiary, and quaternary structures – denaturation destroys the normal folding of the protein, making it inactive. When a protein is denatured, it often coagulates, forming a visible solid. Some denaturing agents include heat, organic solvents, agitation, acid or base, and heavy metal ions.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Wear your safety goggles.

Nitric acid is very corrosive – avoid skin contact with it. If skin contact occurs, flood the area with plenty of running water for 10 minutes.

Heavy metal ions such as Pb^{2+} are poisonous. Avoid skin contact with solutions containing these ions and with the lead acetate paper. Wash your hands thoroughly before leaving the lab.

Ninhydrin spray causes stains.

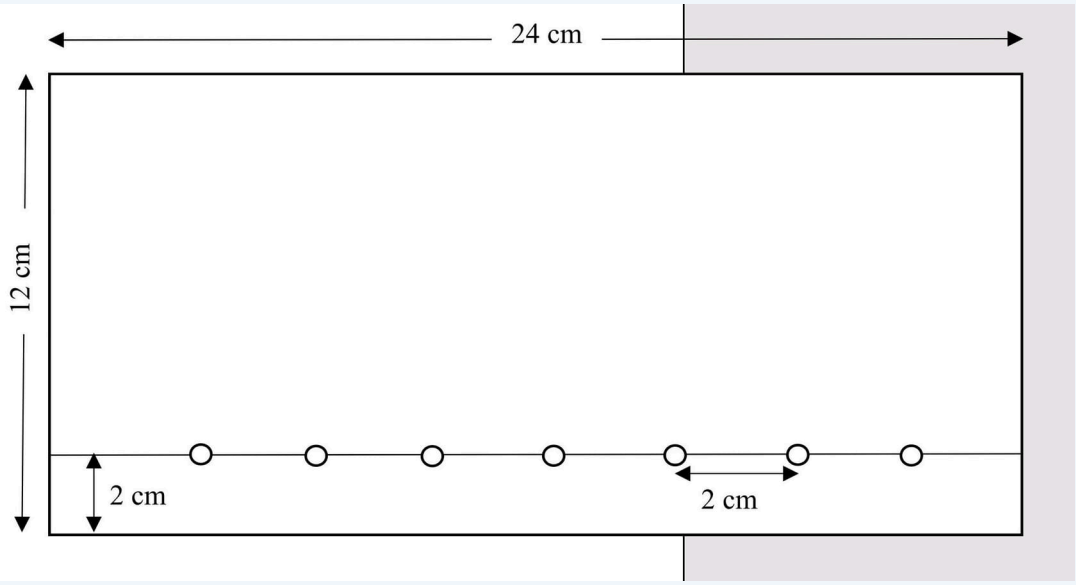
Waste Disposal:

At the end of the experiment, all liquid wastes go into the inorganic waste container. Solid wastes go into the solid waste container.

Section 3: Procedure

Part I. Paper Chromatography

1. Put on a pair of plastic gloves. Pick up a piece of chromatography paper that has been cut to a size of 12 cm × 24 cm. (Do not touch the paper with your fingers. There are traces of amino acids on your skin.) Roll the paper into a cylinder and make sure that it will fit inside a large beaker (400-mL or 600-mL) without touching the sides of the beaker. If it does not fit, cut it to size with a pair of scissors.	

2. Unroll the paper and use a pencil (not a pen!) to draw a line across the paper 2 cm from the edge of the paper. This will be the starting point for the amino acids (this is also called the **origin line**). With the pencil, mark seven points along this line, making sure that they are at least 2 cm apart and at least 2 cm from either edge of the paper. *Note: The illustration below is not drawn to scale.*  	
3. Using a toothpick or a capillary tube, apply a small spot of each amino acid solution to the marked spots on the paper. You will test **phenylalanine, alanine, glutamic acid, serine, lysine, aspartic acid, and an unknown amino acid**. Use a different applicator for each amino acid and be careful not to mix them up. (If the amino acid solutions get contaminated with traces of other amino acids, your results will be inconclusive.) Apply a small spot by lightly touching the applicator to the paper. Let the spot dry and repeat the process on the same area to apply more of the same amino acid. Keep the diameter of the spot as small as possible. Label each spot with a pencil as you go along. Allow the spots to dry.	Identification code for the unknown amino acid: Unknown # _____
4. Take a 600-mL or 400-mL beaker to the hood. Label it with your name. Pour some of the chromatography solvent into the beaker until it reaches a depth of about 1 cm. The liquid level must be below the origin line on your chromatography paper. Cover the beaker tightly with plastic wrap and leave it in the hood.	
5. Roll your chromatography paper into a cylinder with the spots and labels facing out. Staple the edges together, but do not let them overlap. Remove the plastic wrap from the beaker. Carefully lower the cylinder into the beaker so that the row of amino acids is on the bottom edge of the paper. Do not let the paper touch the sides of the beaker. Cover the beaker with plastic wrap and leave it where it is in the hood. Do not move the beaker. Let the solvent rise until it is 2-3 cm from the top of the paper. (This may take 45-60 minutes.) Do not let the solvent reach the top of the paper. While you are waiting, go on to Part 2 of the experiment.	
6. When the solvent has risen to within 2-3 cm from the top of the paper, remove the paper from the beaker, remove the staples, and spread it on a paper towel to dry. Immediately mark the **solvent line** with a pencil. Let the paper dry completely.	
7. If you are done with the chromatography, dispose of the solvent in the waste solvent container.	

8. When the paper is completely dry, take it to the hood and spray it evenly with a small amount of ninhydrin solution. **Important**: Use the ninhydrin spray in the hood. Do not breathe the fumes or get the spray on your skin. Let the paper dry. Purple spots should appear which indicate the location of the amino acids on the paper.	
9. Measure the distance between the origin line and the solvent line. This distance is the **distance traveled by the solvent**.	
10. Draw a dot in pencil at the center of each amino acid spot. Measure the distance between the origin line and the center of each spot. This is the **distance traveled by the amino acid**.	
11. Calculate the $\mathbf{R_f}$ value for each of the spots.	
12. Determine the identity of the unknown by comparing its R_f value and color to those of the other spots.	

Amino Acid	**Distance traveled by amino acid (cm)**	**Distance traveled**
Phenylalanine		
Alanine		
Glutamic acid		
Serine		
Lysine		
Aspartic acid		
Unknown amino acid		

Identification of Unknown

Unknown number	**Identity of Unknown**	**Reasoning**

Part 2. Chemical Tests for Amino Acids

Note: Be sure to shake the protein solutions before obtaining your sample because some of the proteins tend to settle out.

Note: Several tests require a hot water bath. Prepare the water bath early so it is available when you need it.

1. **pH Test**

Obtain solutions of glutamic acid, lysine, and serine. One by one, stir the amino acid sample with a glass stirring rod and then touch it to a piece of pH paper (not litmus paper). Note and record the pH (by comparing the color of the spot to the pH scale on the box of pH paper - be sure to look at both sides of the box). Make sure to rinse and wipe off the stirring rod between solutions so that you don't accidentally contaminate them.

Observations:

Amino Acid	**pH**
Glutamic acid	
Lysine	
Serine	

2. Biuret Test

In this part, you will test solutions of egg albumin, gelatin, casein, glycine, and proline. Put 2 mL of each solution to be tested in its own test tube. Add 3 mL of 10% NaOH to each tube and mix well. (Remember, if you are using a stirring rod, rinse it and wipe it off between solutions so that you don't contaminate the solutions.) Add 2 drops of 2% copper sulfate ($CuSO_4$) solution (which is blue) to each tube and mix well. Record your observations on the data table below.

3. Xanthoproteic Test

You will test solutions of egg albumin and tyrosine. Put about 1 mL of each solution to be tested in its own test tube. In the hood, carefully add 5 drops of concentrated nitric acid (HNO_3). Caution: nitric acid must be kept under the hood at all times. Avoid all skin contact with nitric acid. Mix each test tube carefully and place the tubes in a boiling water bath for two minutes. Observe and record the color of the solutions. Remove them from the water bath and let them cool. When they are cool, add 10% NaOH to each tube dropwise until the solutions are basic. (You can do this by adding one drop of NaOH, mixing well with a stirring rod, and touching the stirring rod to a piece of red litmus paper. If the litmus paper does not turn blue, add another drop of NaOH to the solution, mix with the stirring rod, and again touch it to a piece of red litmus paper. Repeat this sequence until the litmus paper turns blue – then your solution is basic. You can use the same piece of litmus paper, if you touch the stirring rod to a different dry section each time.) Observe and record any color changes.

4. Ninhydrin Test

You will test solutions of glycine, proline, and egg albumin. Put 2 mL of each solution to be tested in separate test tubes. Add 1 mL of 1 % ninhydrin solution to each tube. Heat the tubes for several minutes in a boiling water bath. Record your observations.

5. Sulfur Test Test solid egg albumin and solid cysteine. Weigh out about 0.2 g of each of these solids and place the samples in separate test tubes. Add 10 mL of 3 M NaOH to each tube and place the tubes in a boiling water bath for 15 minutes. If the solutions start to foam, remove them from the water bath briefly. After 15 minutes of heating, remove the tubes from the water bath and let them cool to room temperature. Add about 10 mL of 3 M HCl to each tube to acidify the contents. Moisten two pieces of lead acetate paper with deionized water and place a piece over the top of each tube. Put the tubes in the boiling water bath again. Do not let the lead acetate paper fall into the solutions. Record your observations. Wash your hands after handling the lead acetate paper, as lead compounds are toxic.	
6. Denaturation Tests a. Put 3 mL of egg albumin solution in a test tube and place the test tube in a boiling water bath. Observe the appearance of the solution before and after heating. b. Put 3 mL of egg albumin solution in a test tube and add 7 mL of 95 % ethanol. Mix well and record your observations. c. Put 3 mL of egg albumin solution in a test tube and add 5-8 drops of $FeCl_3$ solution. Mix well and record your observations. d. Put 3 mL of egg albumin solution in a test tube and add 5-8 drops of 0.2 M $ZnCl_2$ solution. Mix well and record your observations. Put the waste in the inorganic waste container in the hood.	

Observations

Biuret Test

Substance	Observations	Is this a positive or a nega
Egg albumin		
Gelatin		
Casein		
Glycine		
Proline		

Xanthoproteic Test

Substance	Initial Color	Color after acid is added	Color after base is
Egg albumin			
Tyrosine			

Ninhydrin Test

Substance	Observations
Glycine	
Proline	
Egg albumin	

Sulfur Test

Substance	Observations	Conclusion
Solid egg albumin		
Solid cysteine		

Denaturation Tests

Substance	Initial appearance	Appearance afte
Egg albumin + heat		
Egg albumin + 95% ethanol		
Egg albumin + $FeCl_3$		

Egg albumin + Hg_2Cl_2		

Post Lab Questions:

1. According to the results of the paper chromatography, which amino acids were most attracted to the solvent? Which amino acids were most attracted to the paper? Explain.

2. According to your answer to question #1, which do you think is more polar, the chromatography solvent or the paper? Explain.

3. If you had completely hydrolyzed the egg albumin before doing the Biuret test on that sample, what results would you expect for the Biuret test? Explain why. Include what color you would expect to see, whether it is a positive or negative test, and what that means.

4. Suggest a reason why alcohol or other disinfectants are often applied to a person's skin before an injection is given (based on something you learned in this lab).

5. Draw the structure of phenylalanine in its regular form and in its zwitterion form.

6. What substances react in the Biuret test?

7. What substances react in the Xanthoproteic test?

8. Suggest a reason why milk is used as an antidote for lead poisoning. Hint: milk contains a lot of protein.

9. Predict the results of each of the following reactions. Include the observed colors and whether it corresponds to a positive or a negative test.

1. The Biuret test on proline
2. The Biuret test on egg albumin
3. The ninhydrin test on lysine
4. The xanthoproteic test on egg albumin
5. The sulfur test on cysteine

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1.30: Experiment_730_Molecular Structure and Isomerism 1_2_1

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 730: Molecular Structure and Isomerism**

Experiment 730: Molecular Structure and Isomerism

Section 1: Purpose and Summary

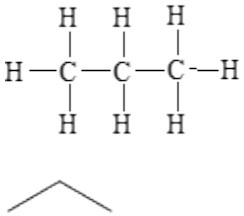
- Construct models of alkanes, haloalkanes, alkenes, simple amino acids and isomeric compounds.
- Use a molecular model kit to construct these molecules and visualize their structure and three-dimensional shape.
- Write structural formulas and name alkanes, haloalkanes, *cis*- and *trans*- isomers and optically active compounds.

Alkanes are saturated hydrocarbons. They contain only carbon and hydrogen and have no multiple (double or triple) bonds present. *Cyclo*alkanes are alkanes containing carbon atoms connected in an enclosed (ring) structure. *Halo*alkanes are molecules that contain carbon, hydrogen, and one or more halogen atoms (F, Cl, Br, or I) as substituents.

Recall that when drawing Lewis structures, a carbon atom will always form four bonds, a hydrogen atom will form one bond and can never be a “central atom”, and a halogen atom will form one bond and have three lone pairs of electrons.

To visualize the three-dimensional structures of organic compounds, it is helpful to build models of the molecules using a molecular model kit. Ball-and-stick model kits contain wooden or plastic balls representing atoms, with a different color for each element, and wooden or plastic sticks that represent bonds. Springs or longer flexible sticks may be used to represent double or triple bonds.

There are various types of formulas that can be written for a particular molecule. The complete **structural formula** shows all the atoms and how they are exactly connected. However, it is somewhat abbreviated and does not give you much three-dimensional information. Atoms are grouped and every bond is not explicitly shown, but the connections between atoms are implied based on how it is written. The **zigzag or line-angle** formula is even more abbreviated: carbon atoms are assumed to be at the ends and at each bend or angle in the structure, and hydrogen atoms are not shown, but implied. The assumption is that each carbon makes four bonds, so if one bond is shown for a particular carbon atom, it is implied that three hydrogen atoms are also bonded to that carbon (to make a total of four bonds). On the other hand, the **molecular formula** does not give any structural information. It just specifies the total number of each type of atom present in the compound. Shown below are various formulas for propane.



$CH_3CH_2CH_3$ C_3H_8

complete structural condensed structural zigzag or line-angle Molecular formula

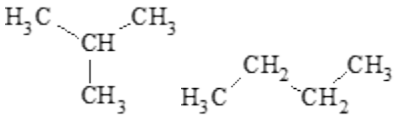
formula formula

The names and formulas of some common alkanes are shown in the table below. See your textbook or lecture notes for the IUPAC guidelines in naming organic compounds.

Name	**Condensed Structural Formula**
Methane	CH_4
Ethane	CH_3CH_3
Propane	$CH_3CH_2CH_3$
Butane	$CH_3CH_2CH_2CH_3$
Pentane	$CH_3CH_2CH_2CH_2CH_3$
Hexane	$CH_3CH_2CH_2CH_2CH_2CH_3$
Heptane	$CH_3CH_2CH_2CH_2CH_2CH_2CH_3$
Octane	$CH_3CH_2CH_2CH_2CH_2CH_2CH_2CH_3$
Nonane	$CH_3CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_3$
Decane	$CH_3CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_3$

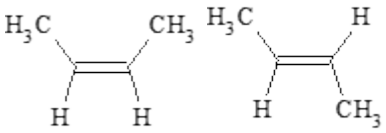
For most organic compounds, if the atoms are arranged differently, a different compound results. Molecules which have the same molecular formulas, but different connectivity of the atoms are called **structural isomers** of each other. Isomers exhibit different properties (such as melting point and boiling point) and have different names.

For example, for butane, (C_4H_{10}), there are two possible ways of arranging the atoms. The first is to have all four carbon atoms connected in an unbroken line. This is called “n-butane”. The second is to have three carbons connected in a sequence with the fourth carbon attached to the middle carbon. The common name for this molecule is “isobutane”. The structures of n-butane and isobutane are shown below. Note that since the two molecules have the same molecular formula but different structures, they are considered isomers of each other.



n-butane isobutane

Another type of isomerism observed in organic compounds is stereoisomerism. Stereoisomers have the same molecular formulas and same connectivity of the atoms, but different spatial arrangement of the atoms. Geometric isomers, a type of stereoisomers, show two substituents in the same side (*cis*) or opposite sides (*trans*) of a carbon-carbon double bond or a ring structure. For example, in 2-butene, $CH_3CH=CHCH_3$, the *cis* isomer has the two methyl groups on the same side of the double bond, while the *trans* isomer has the two methyl groups on the opposite sides of the double bond.



cis-2-butene *trans*-2-butene

Another type of stereoisomers are optical isomers where substituents on a tetrahedral carbon (stereocenter) are arranged in such a manner that the mirror images of the molecular structure are non-superimposable. They are also called *enantiomers*. Optical isomers are common in biological systems. There is a standard naming convention for them. The R ('right-handed') configuration and the S ('left-handed') configuration which depend on the priority rules on the groups bonded to the stereocenter.

Section 2: Procedure

Use your molecular model kit to construct a three-dimensional model of each molecule in this experiment.

The following is a quick guide to using your molecular model kit:

- Each colored center corresponds to a specific atom.
- Refer to the color key provided in the kit, but generally the color scheme is as follows:

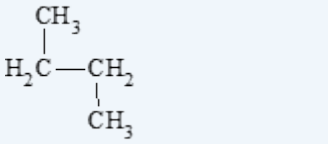
1. Black is for Carbon
2. White is for Hydrogen
3. Red is for Oxygen
4. Blue is for Nitrogen
5. Green/Orange/Purple is for a halogen
6. Yellow is for sulfur

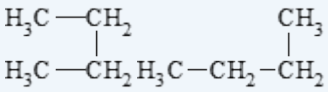
- Use the medium length gray links for single bonds.
- Use the short white links for lone pairs.
- Use the flexible long gray links for making double or triple bonds.

Part 1: Alkanes

1. Construct a model and sketch a 3-D representation for methane (CH_4). Complete the worksheet below (starting in Section 3) by writing the complete structural formula and the condensed structural formula for methane.
2. Repeat step 2 for ethane and propane. Notice that all the bond angles are equivalent for each molecule. From the Valence Shell Electron Pair Repulsion (VSEPR) model, the bond angle for all carbon atoms is predicted to be 109.5o and the geometry is tetrahedral.
3. Repeat step 2 for butane. Notice that in the 3-D structure, the carbons are not actually arranged in a straight line.

4. Using your model for butane, rotate the bonds and twist the molecule. There is free rotation around single bonds, so molecules are constantly twisting, rotating, and changing their **conformation**. It is important to understand that this type of motion is happening constantly in any given molecule. By looking at the following structures, you should be able to recognize that they are equivalent and represent the same molecule. They are NOT isomers of each other!





5. Construct a model for isobutane. (The IUPAC name for isobutane is 2-methylpropane.) Compare this with your model for butane from step 5. Note that butane and isobutane cannot be interconverted without breaking the bonds. Thus, they represent distinct molecules with different properties. Draw the complete structural formula and the condensed structural formula for isobutane in the worksheet below.

6. Locate the chemistry handbook in the lab. Look up the molar mass, melting point, boiling point, and density for butane and for isobutane (which might be listed as 2-methylpropane). Record the data in the worksheet below.

7. Now, consider pentane, C_5H_{12}. There are three isomers for pentane. Construct a model for each isomer. For each isomer, write the condensed structural formula and IUPAC name. Look up the molar mass, melting point, boiling point, and density for each isomer and record below.

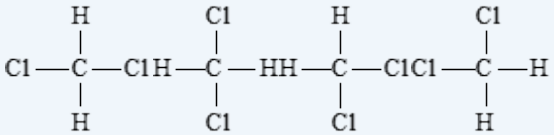
Part 2: Cycloalkanes

Construct models of cyclopropane, cyclobutane, and cyclopentane. You will need to use flexible sticks for the carbon-carbon bonds in the first two molecules because their bond angles are significantly different from the normal 109.5°. Cyclopropane and cyclobutane have a significant amount of “ring strain.” Draw the complete structural formula, the condensed structural formula, and the line-angle formula for each. Record your data in the worksheet below.

Part 3: Haloalkanes

1. Starting with a model of methane (CH_4), replace one hydrogen atom with a chlorine (green) atom. You have made a model of CH_3Cl (chloromethane). Does it matter which hydrogen is replaced? All hydrogen atoms in CH_4 are considered equivalent. Each one has the same angle and position relative to the other hydrogen atoms in the molecule. Each one is equidistant from all the others.

2. Again, starting with a model of CH_4, replace two hydrogen atoms with chlorine atoms. Are there different isomers possible for this formula? Given the structures below, are these molecules isomers or equivalent? Explain.



3. Go back to your model of ethane, CH_3CH_3. Are all the hydrogen atoms equivalent? Explain.

4. Starting with your model of ethane, make a model of chloroethane, CH_3CH_2Cl. Again, do this by removing one of the hydrogen atoms and replacing it with a chlorine atom. Are there different isomers possible for this molecule? Explain. Draw the three-dimensional structure of this molecule.

5. From your model in step 4, replace another hydrogen atom with a chlorine atom. There are two possible locations for the second chlorine atom: on the same carbon as the first chlorine atom, or on the other carbon atom. Thus, there are two isomers possible for the molecular formula $C_2H_4Cl_2$. Make models of both isomers and compare them.

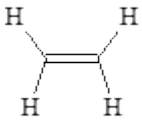
Notes: There are only two isomers possible for this molecule. There are five possible locations for the second chlorine atom. But since two of the positions are equivalent to each other (free rotation around the bonds), placing the chlorine at either of these two positions results in the same molecule. Similarly, the other three positions are equivalent to each other, so placing the chlorine at any of these positions will give you the same molecule. Convince yourself of this by making various models of the different possibilities and noticing which ones are the same. Even though there are five possible locations for the chlorine, there are only two types of positions, hence, only two isomers are possible.

6. Draw the condensed structural and line-angle formulas for the two isomers and give their IUPAC names in the worksheet below.

7. How many isomers are possible for the molecular formula $C_3H_6Cl_2$? Draw and name each isomer in the worksheet below. Make models of the molecules. Make sure that all the isomers you list are different molecules.

Part 4: Geometric isomerism

1. Starting with a model of ethane CH_3CH_3. Remove two of the hydrogens. Replace the hydrogens with a second bond between the two carbons using one of the longer bonds in the model kit. This new compound is called ethylene. It is sometimes written as $H_2C{=}CH_2$. It has a carbon-carbon double-bond.



2. Remove a hydrogen and replace it with a chlorine. This formula is CH_2CHCl and is called chloroethylene. The double bond restricts where the chlorine can be and since the molecule cannot be turned around in any geometric position, there is only one way that chloroethylene can be constructed.

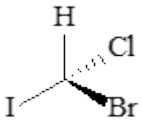
3. There are 3 hydrogens remaining on chloroethylene. Remove the hydrogen from the carbon that already has one chlorine attached to it and replace it with chlorine. This molecule is called 1,1-dichloroethylene and can also be written CH_2CCl_2. There is only one way to construct this molecule, and the 1's indicate that the two atoms of chlorine are bonded to the same carbon.

4. Swap one of your chlorines with a hydrogen from the second carbon in 1,1-dichloroethylene. There are two possible ways to construct this molecule. Make models of the isomers and compare them. Which one is *cis* and which one is *trans*?

5. Construct models of the *cis* and *trans* isomers of 2-pentene. See how these two compounds are geometrically bonded differently. They have slightly different chemical properties and chemical reactivity. Draw their structures in the worksheet below.

Part 5: Optically active centers

1. Starting with a model of CH4, replace the 1st hydrogen atom with a chlorine atom, a 2nd hydrogen with a bromine atom, and a 3rd hydrogen with an iodine atom. The formula for this is CHClBrI.



2. Construct a second model of CHClBrI that is identical to the first one. You should be able to see that the two models can be superimposed on one another.

3. With one of your models of CHClBrI, swap any two of the atoms. For example, the H and Cl can be exchanged with one another but do not change the Br and I.

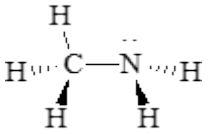
4. Compare your two models of CHClBrI. Note that these compounds are now different. It is impossible to superimpose one upon the other. They are, in effect, mirror images of each other relative to the central carbon atom.

Notes: There are only two isomers possible for this molecule. Convince yourself of this by making various models of the different possibilities and noticing which ones are the same. Even though there are four different atoms attached to the central carbon, only two isomers are possible.

5. Look down the bond that has the highest priority. In CHClBrI, this would be the carbon-iodine bond (carbon is bonded to the element with the highest atomic number). Assign priority to the remaining 3 bonds. In this case, the order would be C-Br > C-Cl >C-H. Draw an arrow in a circle around the 3 bonds. If the arrow is clockwise, it is an *R*- configuration. If it is counterclockwise, it is an S- configuration. Assign the *R*- and S- configuration to your two isomers.

Part 6: Amino acids.

1. Nitrogen bonds similarly to carbon, but it has an additional lone pair of electrons. This lone pair behaves just like a bonding atom—it occupies space at this tetrahedron. Start with a methane, CH_4. Remove a hydrogen and replace it with a nitrogen, N. Add two more hydrogens on the nitrogen. You now have made a model of nitromethane, CH_3NH_2. Notice how the lone pair of electrons on the nitrogen occupies a bonding site and affects the geometry of the molecule.



2. Oxygen had two lone pairs of electrons. Make acetic acid, CH_3COOH. In this case, one of the oxygens is double-bonded to a carbon (like the double-bond formed in part 4, but with two different atoms. The other oxygen is single-bonded to the carbon and has a terminal hydrogen.

3. Amino acids have a general formula of $H_2NCHRCOOH$, where R may be different for different amino acids. The simplest amino acid is glycine, H_2NCH_2COOH. Using your model kit, build glycine. Note that glycine does not have an optically active center (there are no carbon centers with four different things bonded to it).

4. Now remove one of the hydrogens off the CH_2- group in glycine and replace it with a CH_3- group. The new amino acid is called alanine, H_2NCHCH_3COOH. It has an optically active center. Determine if this is an *R*- or S- version that your model represents.

5. Remove one of the hydrogens from the methyl group you just added and replace it with a benzene ring (C_6H_5-). This amino acid is called phenylalanine, $H_2NCH(CH_2C_6H_5)COOH$. Assign the *R*- and S- configuration to your structure.

Section 3: Worksheet

Part 1: Alkanes

1. **CH_4**

3D sketch:	Complete structur Condensed structu

2. **C_2H_6**

3D sketch:	Complete structur Condensed struct

3. **C_3H_8**

3D sketch:	Complete struc Condensed str

4. **butane (C_4H_{10})**

3D sketch:	Complete str Condensed s
Molar mass: Melting point: Boiling point: Density:	

5. **isobutane (C_4H_{10})**

3D sketch:	Complete structu Condensed struc
Molar mass: Melting point: Boiling point: Density:	

6. **Isomers of pentane, C_5H_{12}**

	Condensed structural formula	**IUPAC name**	**Molar mass**
Isomer #1			
Isomer #2			
Isomer #3			

Part 2: Cycloalkanes

	Complete structural formula	**Condensed structural formula**
cyclopropane		
cyclobutene		

cyclopentane		

Part 3: Haloalkanes

1. **Isomers of $C_2H_4Cl_2$**

	IUPAC Name	**Condensed structural formula**
Isomer #1		
Isomer #2		

2. **Isomers of $C_3H_6Cl_2$**

Part 4: Geometric Isomers

Isomers of 2-pentene

	IUPAC Name	**Structural or line-angle formula**
Isomer #1		
Isomer #2		

Part 5: Optically active isomers

Isomers of CHClBrI (Draw bow-tie structures.)

Isomer #1	Isomer #2

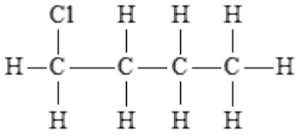
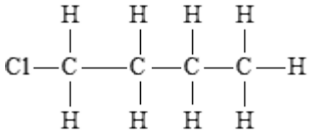
Part 6: Amino Acids

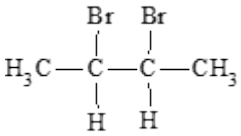
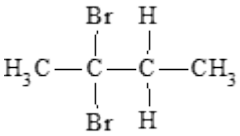
Draw bow-tie structures for each compound.

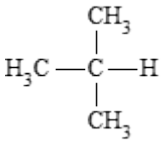
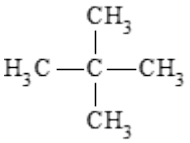
Alanine	Phenylalanine

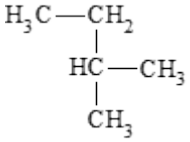
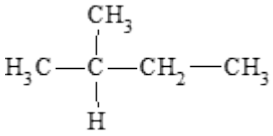
Post Lab Questions:

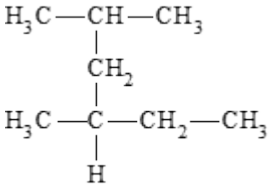
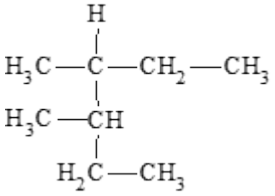
1. For each of the following pairs of molecules, state whether they are isomers, identical, or neither. Briefly explain your reasoning in each case.

1.  and 

2.  and 

3.  and 

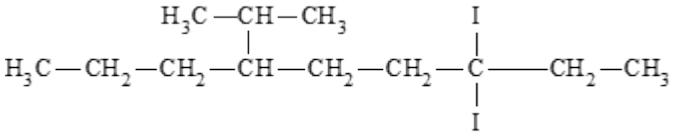
4.  and 

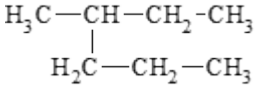
5.  and 

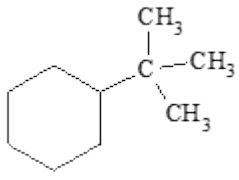
6. and 

2. Name each of the molecules in question #1 above.

3. Name each of the following molecules.

1. 

2. 

3. 

4. Draw condensed structural formulas for:

1. 3-ethylpentane c) 1,2-dibromobutane

2. 1-bromo-3-chlorocyclopentane d) methylcyclohexane

5. Write the condensed structural formulas for all isomers of C_4H_9Br. Name each isomer.

6. Write the condensed structural formulas for the *cis* and *trans* isomers of 1-chloro-2-methyl-2-butene.

7. Draw the enantiomers of 1-bromo-2-chloropropane. Identify the *R*- and *S*- isomers.

Notes:

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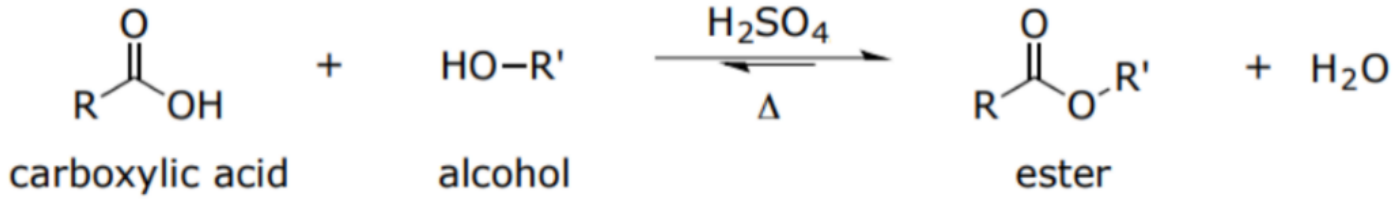
1.31: Experiment_731_Esters _1_0

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 731: Esters**

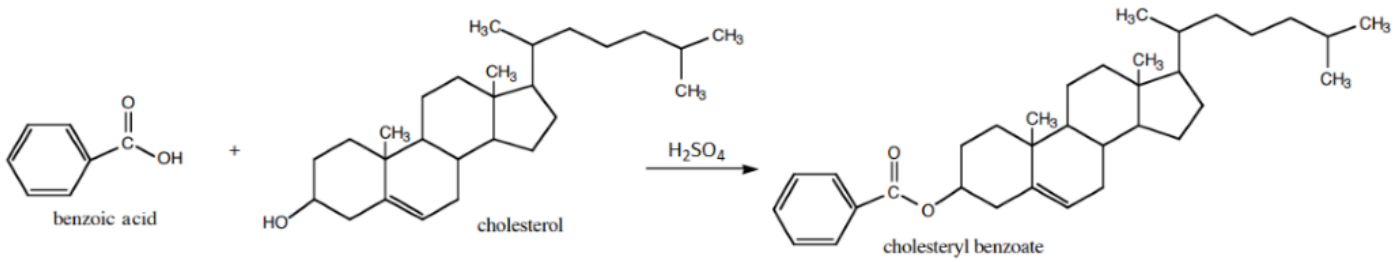
Experiment 731: Esters

Section 1: Purpose and Summary of experiment

Many molecules containing the ester functional group have pleasant, sometimes fruity, fragrances. The synthesis of an ester can be accomplished in one of several ways. In this lab, esterification occurs when an alcohol and a carboxylic acid are reacted in the presence of an acid catalyst, such as concentrated sulfuric acid.



Example of an esterification reaction:



Other synthetic pathways to esters also exist. Acid chlorides react with alcohols to yield an ester and hydrochloric acid. A small amount of pyridine or other base is usually added to the reaction mixture to neutralize the resulting acid. Acid anhydrides can also react with alcohols to produce esters.

Artificial flavors often contain esters. In this lab, isoamyl acetate (banana) and methyl salicylate (wintergreen) work best but also try others to see if you like their odors. Sometimes; however, the carboxylic acids and alcohols may not react completely, and these compounds may contaminate the reaction products such that their foul odors may mask the fragrances of the esters.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Wear your safety goggles.

Sulfuric acid can cause chemical burns that may seriously damage skin and/or eyes. Wear goggles and gloves and avoid contact. If contact occurs, wash with copious amounts of water. A paste made with baking soda (sodium bicarbonate) and water can also be applied to the skin to neutralize the acid.

The carboxylic acids and alcohols used in this experiment can be irritating with foul odors. Keep all reagents in the fume hood to minimize odors.

Waste Disposal:

The residual amounts of amounts of organic compounds used in this lab are safe to dispose down the drain. However, anything larger than 1 milliliter should be captured in the liquid organic waste stream.

Tip: After rinsing the sink with water, check for the pleasant odors of the esters that might remain in the sink!

Pre-Lab Questions:

1) What precautions are necessary when:

A) using sulfuric acid

B) using a warm-water bath

C) smelling the odor of esters

2) What is the purpose of sulfuric acid in today's experiment?

3) Why should test tubes be dry in today's reactions? (Hint: Esterification is an equilibrium reaction.)

4) Butyl alcohol ($CH_3CH_2CH_2CH_2OH$) reacts with acetic acid (CH_3CO_2H) to form an ester.

A) Draw a balanced equation for this reaction.

B) What is the name of this ester?

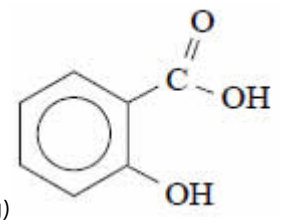
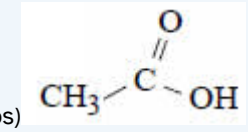
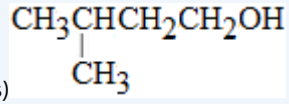
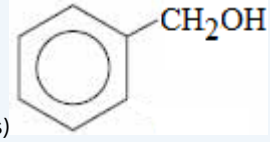
Section 3: Procedure

Part 1: Synthesize the Esters

1. Set up a warm water bath by obtaining a 250 mL or larger beaker and filling it half-full with tap water. Set the beaker on a hot plate and heat the water to 60-65^{o}C.
2. Obtain 6 DRY (water will hinder the reaction), small test tubes. The small test tubes are the ones that contain a volume of about 10 ml. Label the test tubes 1-6.
3. Choose 6 esters to make from **Table 1**.
4. On the DATA SHEET (below) write down the ester that you will prepare in each test tube.
5. Using the quantities listed in **Table 1**, add the listed amounts of each carboxylic acid and each alcohol to the appropriate test tubes.
6. Carefully add 5 drops of concentrated sulfuric acid to each test tube.
7. Heat all the test tubes together in the warm water bath (60-65^{o}C) for 10 minutes or longer.
8. Be sure to monitor the temperature to maintain it within the specified range.
9. Using a test tube clamp, remove the test tubes from the warm water bath and place them in a test tube rack to cool.
10. When cooled to room temperature, use a plastic dropper to add 2 mL (about 40 drops) or more of laboratory water to each test tube and mix well. Two layers will form (like oil and water). The ester will be in the top layer.
11. Use a pipet to remove a few drops of the top layer. Transfer the drops to a clean watch glass and carefully waft (use your hand to fan the odors towards your nose) to smell the ester.

12. Record a description of the actual odor on the DATA SHEET (below). If the odor does not match the odor listed, describe the odor as best you can. (It is possible that the ester may be contaminated by the odors of the carboxylic acid or alcohol!)

Table 1 Synthesis and Characteristics of Esters ***(choose 6)*** ester

Carboxylic Acid	*Alcohol*	*Ester*	*Odor*
Salicylic acid  (0.1 g)	Methyl alcohol (10 drops) CH_3OH	Methyl salicylate	wintergreen
Acetic acid (10 drops) 	Isoamyl alcohol (3-methyl-1-butanol) (20 drops) 	Isoamyl acetate	banana
Acetic acid (8 drops)	Octyl alcohol (20 drops) $CH_3(CH_2)_6CH_2OH$	Octyl acetate	orange
Acetic acid (10 drops)	Propyl alcohol (10 drops) $CH_3CH_2CH_2OH$	Propyl acetate	pear
Acetic acid (10 drops)	Ethyl alcohol (10 drops)	Ethyl acetate	nail polish remover
Acetic acid (10 drops)	Benzyl alcohol (10 drops) 	Benzyl acetate	jasmine

Propanoic acid (10 drops)	2-methyl-1-propanol (older name is isobutyl alcohol) (10 drops)	Isobutyl propionate	fruity rum smell

Part 2: Identify an Unknown Ester

1. Obtain an unknown ester. If it is labeled with a number or code, record the number or code on the DATA SHEET (below).
2. Use a pipet to remove a few drops of the unknown ester. Transfer the drops to a clean watch glass and carefully waft (use your hand to fan the odors towards your nose) to smell the ester.
3. Record a description of the actual odor on the DATA SHEET (below).
4. Use the odors listed in **Table 1** to identify the unknown ester. Write the name of the ester on the DATA SHEET (below).
5. Return the unused portion of the unknown ester.

Part 3: Clean up (and re-analyze the esters)

1. Empty each test tube one-at-a-time in the sink and rinse with plenty of water. Waft the odors from the sink for a second chance to analyze the odor of each ester.

Data Sheet: Lab group Member(s) _____

Test Tube Number	*Name of Ester*	*Odor listed in Table 1*	*Actual Odor*
1			
2			
3			
4			
5			
6			
Unknown Ester (List number or code here)			

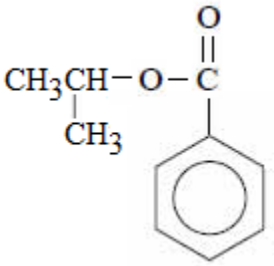
Post-Lab Questions:

1) A student reacts benzyl alcohol with salicylic acid.

A) What is the name of the ester product?

B) Draw the structure of the ester product.

2) Another student made isopropyl benzoate (structure shown here).



A) Draw the structures of the carboxylic acid and alcohol used to synthesize isopropyl benzoate.

B) Write the names of the carboxylic acid and alcohol underneath your drawings.

3) Draw balanced equations for the reactions that took place in each of your 6 test tubes from today's lab.

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1.32: LMC Experiment_605_Hydrates

Student Name		Laboratory Date: Date Report Submitted:	_____
Student ID		Experiment Number and Title	**Experiment 605: Hydrates**

Experiment 605: Hydrates

Section 1: Purpose and Summary

- Determine the percent water of hydration in a hydrate sample.
- Determine the number of moles of water, *x*, per mole of anhydrous salt and write the chemical formula of the hydrate sample.

Hydrates are ionic compounds that contain water molecules as part of their crystal structure. The bound water is called the water of hydration. Some compounds lose this water of hydration spontaneously (efflorescent) while some may require heating. On the other hand, an anhydrous salt (without water) can absorb water from the atmosphere and spontaneously dissolve in its own water of hydration (deliquescent).

A hydrate contains a definite number of water molecules bound to each ionic compound or *anhydrous salt*. In this experiment, students will dehydrate an unknown hydrate sample by heating, and calculate the amount of water lost in the process. Students will determine the ratio between the moles of water lost and the moles of anhydrous salt and write the chemical formula of the hydrate sample.

Section 2: Safety Precautions and Waste Disposal

Safety Precautions:

Use of eye protection is recommended for all experimental procedures.

A hot crucible will break if placed directly on a cold surface. Set hot crucibles on to wire screens to cool.

Waste Disposal:

When you are finished with the experiment, discard solid waste in the container marked ‘solid waste’ in the fume hood.

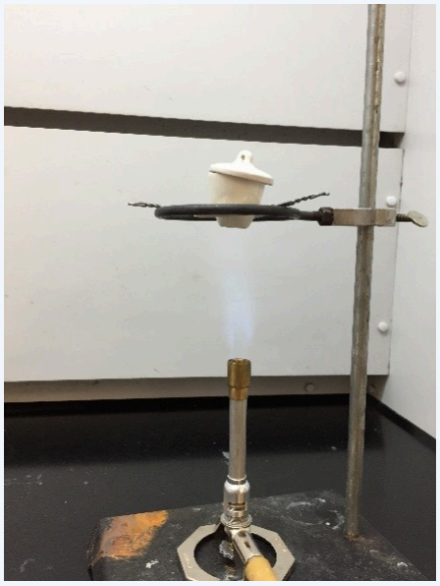
Section 3: Procedure

Part 1: Deliquescence and Efflorescence

Prepare two clean and dry watch glasses. Transfer a few crystals of sodium sulfate decahydrate, $Na_2SO_4{\times}10H_2O$, into one watch glass, and anhydrous calcium chloride, $CaCl_2$, into the other. Observe each sample occasionally as you perform the rest of this experiment. Record your observations below.	
$Na_2SO_4{\times}10H_2O$	**$CaCl_2$**

Part 2: Formula of an unknown hydrate

Possible unknowns: $CaCl_2 \times xH_2O$, $CaSO_4 \times xH_2O$, $AlK(SO_4)_2 \times xH_2O$, $MgSO_4 \times xH_2O$

1. Obtain an unknown hydrate from your instructor. Record identification code for your unknown.	Identification code for unknown:
2. Assemble a wire triangle supported on an iron ring attached to a ring stand. Put a clean, dry porcelain crucible and lid on the wire triangle and heat over a Bunsen burner flame for about three (3) minutes to ensure complete dryness. An example setup is shown:  Allow the crucible to cool on the wire triangle. When the crucible is cool and safe to touch, weigh on an analytical balance. Record exact mass of the crucible and lid.	Mass of crucible and lid: 1. _____ grams
3. Transfer 2 – 4 grams of your unknown sample into the crucible and weigh again. Record exact mass. NOTE: You need to have both the total mass of the crucible, lid and sample as well as just the sample. You can determine the mass of the sample by subtraction of the mass of the crucible (a).	Mass of crucible, lid and sample (before heating): 2. _____ grams
4. Put the crucible and sample back on the wire triangle. Position the crucible such that it is at a slight angle on the triangle. Place the crucible lid so that the lid is slightly ajar. Gently heat the crucible for 5 minutes over a Bunsen burner flame (slowly move the burner back and forth across the bottom of the crucible).	
5. After gentle heating for about 5 minutes, increase the heat applied to the crucible, lid, and sample until the bottom of the crucible turns red. Continue providing high heat for additional 10 minutes. Let the crucible cool for 5 to 10 minutes. Using crucible tongs, place the lid and the crucible on a wire gauze on the bench to finish cooling to room temperature.	

6. When the crucible is cool and safe to touch, weigh on an analytical balance. Record exact mass. Be sure to include the lid with the crucible on the balance.	Mass of crucible, lid and sample (after final heating): 3. _____ grams
7. To ensure complete dehydration of sample, reheat the crucible, lid, and sample as in step #5, except to heat it for 5 minutes. Cool, and weigh again. Record exact mass. (*Repeat this process, if necessary, until the mass of the sample is within 0.01 g of the previously recorded mass.*)	Mass of crucible, lid and sample (after 2nd, etc. final heating): 4. _____ grams

Part 3: Reversibility of hydration

1. Transfer a small amount of solid copper (II) sulfate pentahydrate, $CuSO_4 \times 5H_2O$, that just fills the bottom of a clean, dry 150-mm (medium size) test tube.
2. Using a test tube holder, grasp the test tube containing the hydrate and heat over a Bunsen burner flame while holding the test tube at a 45^o angle.
3. While heating, closely observe the solid and the inside wall of the test tube. Record your observations.
4. When the solid residue seems to be completely dehydrated, allow the test tube to cool completely. Then, add a few drops of laboratory water to the solid in the test tube. What changes did you see? Record your observations.

Appearance of $CuSO_4.5H_2O$:

Appearance of solid after heating:

Appearance of the inside wall of the test tube after heating:

Appearance of solid residue after adding a few drops of laboratory water:

<u>Section 4: Calculations</u>

1. From the masses you recorded in Part 2 of this experiment, calculate the mass of the unknown hydrate sample. **(b) – (a)**	Mass of the unknown hydrate sample: 5. _____ grams

2. Calculate the mass of the heated (dehydrated) sample. This is also called the *anhydrous salt*. **(d) – (a)**	Mass of the heated sample (dehydrated): 6. _____ grams
3. From the mass of the hydrate and the mass of the heated sample, calculate the mass of water lost. **(e) – (f)**	Mass of water lost: 7. _____ grams
4. Calculate percent water in the hydrate. % water $= \frac{\text{mass of water lost } (g)}{\text{mass of hydrate } (e)}$ x 100	***Percent water in the hydrate:***
5. Ask for the identity of the unknown hydrate sample from your instructor. Refer to a periodic table to obtain the molar mass of the anhydrous salt. Then, using this information and the mass of the heated sample, calculate the number of moles of the anhydrous salt. Identify of the anhydrous salt: _____ Molar mass of anhydrous salt: _____g/mol Show your equation here:	Moles of anhydrous salt: 8. _____ mol
6. Similarly, determine the molar mass of water. Then, calculate the number of moles of water lost from the sample. Molar mass of water, H_2O: _____g/mol Show your equation here:	Moles of water lost: 9. _____ mol
7. Calculate the ratio of moles of water lost to moles of anhydrous salt. **(i) ¸ (h)**	mole of H_2Omole of anhydrous salt ratiomole of H_2Omole of anhydrous salt ratio 10. ___ : ___
8. The ratio calculated **(j)** is expressed in the formula of the compound (hydrate). Write the chemical formula of the hydrated form of your unknown sample. *For example*, if a given amount of hydrated copper(II) sulfate gave off 0.060 mole of H_2O and left behind 0.012 mole of anhydrous copper(II) sulfate, $CuSO_4$, then the ratio of H_2O to $CuSO_4$ is 5:1, and the formula would be written as $CuSO_4 \times \mathbf{5}H_2O$.	**Chemical formula of the unknown sample:**

Post-lab Questions

1. Calculate the mass percent of water for the hydrate, $LiNO_3 \times 3H_2O$.

2. What will be the probable effect if you kept the crucible completely covered during the entire heating and cooling processes? Would your calculated percent water in the hydrate be high, low, or unaffected? Explain.

If 2.752 g sample of $Ca(NO_3)_2 \times xH_2O$ is heated to constant mass, the residue weighs 1.941 g. Determine the value of *x* and the formula of the hydrate.

Notes:

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CHAPTER 2: TECHNIQUES

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2.1: Technique A Laboratory Notebook_1_2_1

Technique A: Use of Laboratory Notebook

Section 1: Purpose of Technique

The laboratory notebook is a permanent record of a researcher's activities. The main purpose of maintaining a laboratory notebook is to preserve experimental plans, study design or protocol, procedures that were followed, observations, results, conclusions, and recommendations. The information that is documented must be done in a way that another scientist can replicate the study based on the information presented in the notebook or binder.

Supplemental quality control activities and practices related to equipment maintenance and calibration are recorded on appropriate forms and maintained in log books as required under other laboratory Standard Operating Procedures. Unless there is a critical operation, it is generally not necessary to record supplemental data twice, though the activities should be referred to and cross-referenced in the laboratory notebook. For example, if the laboratory water supply is monitored, measured and recorded in a separate logbook, then you do not generally need to also record that monitoring result in your laboratory notebook.

Section 2: How to Start Your Laboratory Notebook

Requirements for a Laboratory Notebook

The laboratory notebook is usually bound with sequentially numbered pages. Several styles are available. Some organizations require acid-free paper for archival purposes. Others require a carbon-copy for immediate archival at the end of the day. Pages can be ruled or graph-ruled. Some examples are provided below:



For most educational settings a bound composition book is sufficient. If the pages are not sequentially numbered, then

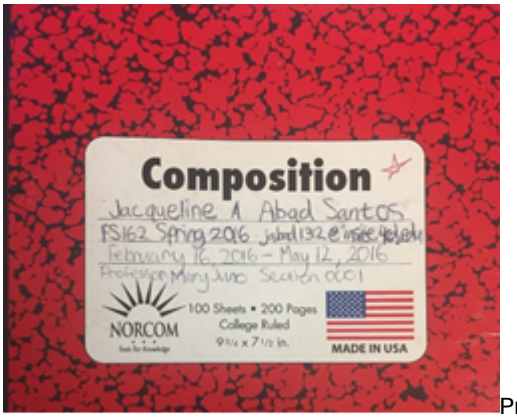
sequences can be established by diligent recording of entry dates:



Laboratory notebooks should be used with either blue or black permanent ink. Many non-black or blue inks fade with time.

Section 3: Components of a Laboratory Notebook

Part 1: Owner's Name, Dates of Use, and Course Title

1. Put your name on the outside cover, inside cover, or on the first page of your notebook to identify that you own this laboratory notebook.
2. Identify what period of time this laboratory notebook is in service. Usually that is a start date on the cover, followed by an end date when the notebook is completed.
3. Record your course title, professor, and section number for reference.
4. 

Provide an email address and contact phone number in case you leave your notebook in a classroom.

Part 2: Table of Contents and Numbered Pages

1. A Table of Contents is optional, but useful for yourself and for other people.
2. Your Table of Contents should have the page number, title of experiment, and dates of when you started and finished your experiment. This makes it easier for others to read your laboratory notebook.
3. Make sure to include enough pages in the front for your table of contents.
4. After making enough space for your table of contents, if your notebook is not sequentially numbered, make sure to do so using black or blue ink. Date and initial all information collected, recorded, and calculated in your notebook at the end of each day of working in the laboratory.

Part 3: Experiment Entries

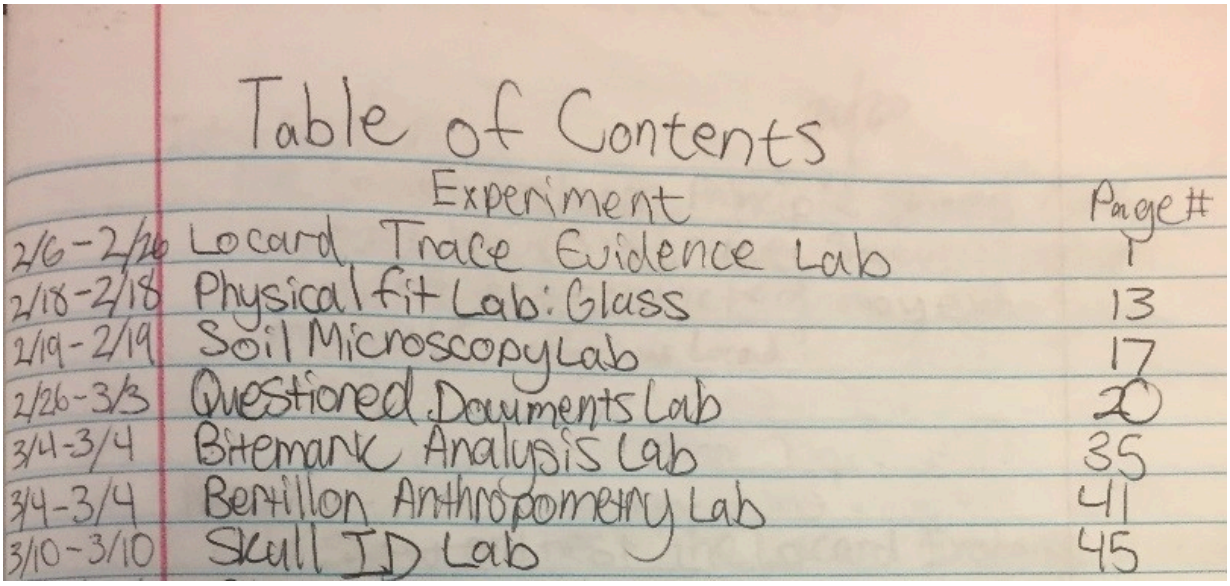


Table of Contents

	Experiment	Page #
2/6-2/26	Locard Trace Evidence Lab	1
2/18-2/18	Physical fit Lab: Glass	13
2/19-2/19	Soil Microscopy Lab	17
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3/4-3/4	Bitemark Analysis Lab	35
3/4-3/4	Bertillon Anthropomerry Lab	41
3/10-3/10	Skull ID Lab	45

1. For each experiment include the day you started it and the title of the experiment at the beginning of the page.

2. Document your daily plan of the experiment before starting it. Include the purpose of the experiment, background information, what techniques you will be using, what equations you are going to be using for your calculations, and what reagents and materials will you be using.

3. Start a section for recording your observations. Write down clear, concise, and detailed descriptions of what you see. Include all that was expected and any observations you did not expect. You may have an idea of what you expect to happen if you have completed your pre-laboratory research and read the background information. Unusual observations should be recorded—these observations may lead to further experimentation. Entries should be grammatically correct, legible and honest.

4. Include your raw data from experiment. You never know what information may be important, so write down as much as you can. You can organize data in tables. Tape or glue in any data you have printed out or recorded on a separate piece of paper. If you use a computer program, record the program name and version number if relevant.

5. After observations, you need a data analysis section. This will include any calculations needed to properly interpret your data.

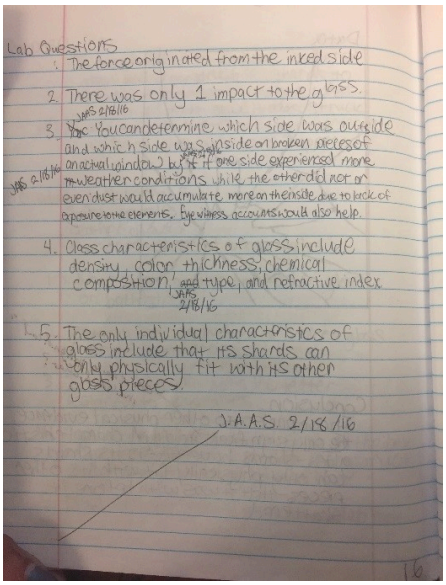
6. Include a conclusions section of what you did well, what you could have done better, and what future scientists can do to improve the results of the experiment.

7. Occasionally, another person may have done some parts of the experiment. Note any parts someone else may have done and have them date and initial the relevant parts of your laboratory notebook.

8. If an experiment is too long for one page, note that the experiment continues on to the next page. If you have to skip pages because you are doing multiple experiments, include references back to the original experiment. So if Experiment 12 ends on page 23 and is continuing on page 35, a note on page 23 should read ‘continued on page 35’ and a note on page 35 should read ‘Experiment 12 (continued from page 23).’

Keeping A Legally Defensible Document

All data goes in the notebook, even outliers, or “bad” data points. A failed experiment should still be documented in the laboratory notebook.



Do not take out any pages or remove any data or observations you have made in your notebook. Do not skip any pages in your laboratory notebook. Cross out any unused portions of your laboratory notebook and mark it with the date and your signature.

Cross out any mistakes with a single line only. Put in corrections without covering anything. Sign and date all your corrections.

To correct a large section, block out with one diagonal line from corner to corner followed by a date, signature, and reason for crossing it out.

Reference

"Standard Operating Procedure for Use and Maintenance of Laboratory Notebooks and Project Binders", U. S. Environmental Protection Agency Office of Pesticide Programs, Microbiology Laboratory, Environmental Science Center, Ft. Meade, MD. SOP Number: ADM-05-04; Date Revised: 06-20-17. https://www.epa.gov/sites/production.../adm-05-04.pdf (Accessed 10/29/2018).

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2.2: Technique B Significant Figures_1_1_1

Technique B: Significant Figures

Section 1: Purpose of Technique

The number of *significant figures* of a quantity is the count of digits required to maintain precision. The number of significant figures is very important. Proper use of significant figures gives an immediate estimate of how precisely the quantity has been measured.

For example, a bathroom scale shows a value of 201.3 pounds for a person's weight. When converting from pounds to kilograms, you divide by a conversion factor of 2.20462 pounds per kilogram. Modern calculators show a result of 91.30825267 kg, but not all of these digits are needed (or desired). You should keep only the digits necessary to describe the precision of the original measurement but in the new units. Using the rules for significant figures and mathematical operations that follow, the result should be reported as 91.31 kg.

Section 2: Rules for Significant Figures

Determine the number of significant figures in a measurement:

1. All nonzero digits are significant.

Examples: 4.79 has three significant figures.

2779 has four significant figures.

856.429 has six significant figures.

2. Zeros in the middle of a number are significant.

Examples: 1.0049 has four significant figures.

4.50809 has six significant figures.

780.12 has five significant figures.

3. Leading zeros (at the very beginning of the number) are not significant.

0.032 has two significant figures. (It may be written as 3.2×10^{-2}).

0.0000411 has three significant figures. (It is also 4.11×10^{-5}.)

If you are not sure of how many significant figures are present, convert the number to scientific notation. Numbers written in scientific notation are in the form of a coefficient multiplied by 10 raised to an exponent. The number of significant figures is the number of digits in the coefficient.

Examples: 0.00579 has three significant figures (may be written as 5.79×10^{-3})

0.048023 has five significant figures (may be written as 4.8023×10^{-2})

0.000024 has two significant figures (may be written as 2.4×10^{-5})

4. Zeros at the end of a number are significant if the number contains a decimal point.

Examples: 1.30 has three significant figures.

4.00 has three significant figures.

350. has three significant figures. (The decimal point at the end is the indication that the zero is significant.)

5. Zeros at the end of a number which does not contain a decimal point may or may not be significant (it is unclear). When in doubt, assume they are not significant.

The number 2500 could have been measured to the nearest ± 100 or to the nearest ± 10 or to the nearest ± 1. Since it is not indicated, you are forced to assume the worst case: the greatest uncertainty. 2500 has two significant figures. (2.5×10^3)

140 has two significant figures. (1.4×10^2)

If you are recording a number such as "2500", it is best to include the error estimate so there is no ambiguity.

Section 3: Rules for Rounding

If a calculated number has too many digits, it needs to be rounded to the appropriate number of significant figures. The rules for rounding are as follows:

1. Increase the last retained digit by one if the next digit is greater than or equal to 5.

Examples: 4.248 rounded to three significant figures. It rounds to 4.25

347.9 rounded to three significant figures. It rounds to 348

7815.246 rounded to six significant figures. It rounds to 7815.25

2. Decrease the last retained digit by one if the next digit is less than 5.

Examples: 94.24571 rounded to three significant figures. It rounds to 94.2

1.50802 rounded to five significant figures. It rounds to 1.5080

624804 rounded to two significant figures. It rounds to 620000

NOTE:

There are several ways of dealing with rounding when the next digit after the last retained digit is 5. The convention we are using is covered by Rule 1 (above). However, your instructor may want you to use a different one.

Section 4: Determining the number of significant figures in a calculation

A calculation cannot be more precise than the least precise measurement used to do the calculation. (The least precise measurement limits the precision of the result.)

1. Multiplication and Division:

The number of significant figures in the result should be the same as that in the number with the least significant figures.

3.42 ÷ 1.3 = 2.630769 Rounded result = 2.6

The reported result of 2.6 has two significant figures. This is because the number "1.3" in the original calculation has 2 significant figures (the fewest in this calculation).

43.10 × 9.22713 = 397.6893 Rounded result = 397.7

The reported result of 397.7 has four significant figures. This is because the number "43.10" in the original calculation has 4 significant figures (the fewest in this calculation).

7.58432 * 18.54 * 6.542 ÷ 42.5 = 21.64452 Rounded result = 21.6

The reported result of 21.6 has three significant figures. This is because the number"42.5" in the original calculation has 3 significant figures (the fewest in this calculation)

2. Addition and Subtraction:

The number of decimal places in the result should be the same as in the number with the fewest decimal places. The digits in **bold** are the important ones to look at.

4.381 31.5563

+131.**4** - 1.41**0**

135.781 30.1463

Round to nearest 0.1 Round to nearest 0.001

Report result as 135.8 Report result as 30.146

3. Logarithms and Exponents:

A logarithm is made of two parts, the *characteristic* and the *mantissa*. The characteristic is everything before the decimal point in a logarithm. The mantissa is everything after the decimal point in a logarithm. Unless the base of the log is specified differently, it is assumed to be the common log of base 10.

Example 1: log (345.2) = 2.5381 which means that $10^{2.5381} = 345.2$

In this example, the number "2" in the logarithm is the characteristic and ".5381" is the mantissa.

Example 2: log (0.0024) = -2.62 or, $10^{-2.62} = 0.0024$

In this example, the number "-2" is the characteristic and ".62" is the mantissa.

With regard to significant figures, the number of significant figures in the mantissa should be the same as the number of significant figures of the original number. In Example 1, "345.2" and ".5381" have the same count of four significant figures. In Example 2, "0.0024" and "0.62" have the same count of two significant figures.

Converting quantities into logarithms is commonly found when working with pH and concentrations. The mathematical definition of pH is: pH = - log (concentration of H^+ in moles per liter). Moles per liter is abbreviated as 'M'.

Example: Determine the pH of a solution containing a concentration of H^+ of 2.56×10^{-4} M, the calculation is:

pH = - log (2.56×10^{-4} M) = - (-3.591760035) which rounds to 3.591

In this example, the original concentration (**2.56** $\times 10^{-4}$ M) has 3 significant figures (shown in **bold**). The mantissa of "3.**591**" has 3 significant figures.

4. Physical Constants, Discrete Numbers, and Mathematical Constants.

Physical Constants

Final results should reflect the uncertainty due to experimental error, not from calculations. Many physical constants have been measured to great precision so you have many more significant figures than you require. For example, Avogadro's Number (6.0221409×10^{23}) or the speed of light (2.99792×10^8 meters/second) have been measured to great precision. If you are using these constants in a calculation, and are hand-entering the numbers in your calculating device, then include at least two more significant figures in the constant than the quantity with the fewest significant figures in your calculation.

Example: If you are calculating how many atoms are present in a mass of *1.25 x* 10^{-5} *grams of lead (Pb)*, using the atomic mass of *207.19 grams/mole*, the calculation should be set up as:

$$\frac{1.25\times10^{-5}\text{grams } Pb}{1} \times \frac{1 \text{ mole } Pb}{207.19 \text{ grams } Pb} \times \frac{6.0221409\times10^{23} \text{ atoms } Pb}{1 \text{ mole } Pb} = 3.63321 \times 10^{16} \text{ atoms } Pb$$

Note that the units cancel except for *atoms Pb*. The rounded result should have three significant figures, so the final result is *3.63 x 10^{16} atoms Pb*.

In this example, the atomic mass of Pb is a reference value presented with five significant figures, two more than the minimum of three significant figures. Avogadro's Number of 6.0221409 x 10^{23} should be entered in the calculation with at least five significant figures (more significant figures will not affect the calculated result after rounding).

Discrete Numbers

Measurements of discrete objects can be considered to have infinite precision. For example, there are precisely 12 eggs in a dozen (i.e., 12.00000...).

Example: If you are calculating a molecular weight that contains 2 copper atoms, the mass of the copper atom (63.55 g/mol) can be multiplied by 2 with no loss of significant figures.

Mathematical Constants

It generally does no harm to use too many significant figures in your constants, but it is not necessary. If your calculator has a constant such as π or *e*, then just use the button on your calculator and the number provided.

Example: The area of a circle is given by A= π r^2. For a radius of 4.247 cm, find the area.

A = 3.14159 × 4.247 cm × 4.247 cm = 56.6648 cm^2. This rounds to 56.66 cm^2.

Note that the value of π used has six significant figures, only two more than is needed for the quantity of “4.247 cm” which has four significant figures.

The use of 3.14 as a value of π in the above calculation would be incorrect because it has too few significant figures. The following calculation is provided to show what happens when you use too few significant figures.

Example of *incorrect* significant figure usage:

A = 3.14 × 4.247 cm × 4.247 cm = 56.6362 cm^2. This rounds to 56.64 cm^2.

Although it seems like a minor difference (0.02 cm^2) between the two numbers, these differences can propagate through many calculations and result with real differences in results.

Section 5: Determining significant figures for measured numbers

Laboratory measuring equipment such as balances, scales, glassware marks, rulers, and calipers have limits on precision. The number of significant figures in measured quantities should imply the precision in the measurement. Equipment with digital readouts can be usually read as-is. All other equipment may have precision limits declared on them, or may be derived from the precision of the smallest graduation.

Refer to the specific users' manual, experiment or technique for additional guidance, or ask your instructor. Some general examples are provided below for common laboratory equipment.

For balances:

A balance that is precise to the nearest 0.01 grams (or 10 milligrams) will have a number precise to the nearest 0.01 grams. A mass measured on this balance as 5.24 g has three significant figures and is assumed to have an error of +/- 0.01 grams.

A balance that is precise to the nearest 0.1 milligrams will have a number precise to the nearest 0.0001 grams. A mass measured on this balance as 5.2400 has five significant figures and is assumed to have an error of +/- 0.0001 grams.

For rulers:

A ruler that is graduated to 0.1 centimeter (or 1 millimeter) will be measured to the nearest 0.1 cm or 1 mm. The length should be estimated to the nearest graduation. A length of 4.2 cm or 42 mm has two significant figures.

For graduated cylinders:

A 10 ml graduated cylinder that is graduated to 0.1 milliliter has a tolerance of 0.01 ml. It should be measured to the nearest 0.01 ml by estimation. A volume measured by this cylinder as 5.62 ml has three significant figures.

For burets:

A 50 ml buret that is calibrated to 0.1 milliliter can be estimated to the nearest 0.01 milliliter if sufficient distance exists between calibration marks. A volume measured as 15.24 ml has four significant figures.

For transfer pipets:

A 25 ml "Class A" transfer pipet has a specification of +/- 0.03 ml. Properly used, this transfer pipet will deliver 25.00 ml of a solution, which should be recorded with four significant figures. Note: A more formal error analysis may use the specific declared error limits and is beyond the scope of this document.

For volumetric flasks:

A 100 ml Class A volumetric flask has a tolerance of +/- 0.08 ml. Properly used, this flask will contain 100.0 ml of a solution, which should be recorded with 4 significant figures. Note: A more formal error analysis may use the specific declared error limits and is beyond the scope of this document.

Section 6: Other ways of reporting significance

Using significant figures is the most common way to present quantities with an estimate of the uncertainty in the last digit. It is a little more complicated to calculate statistically valid error range estimates.

However, sometimes specific error estimates are presented along with the number. Here are some results from metals analysis of a National Institute of Standards and Technology (NIST) standard reference material (Buffalo River Sediment, Standard Reference Material 2704):

Selected element	Weight %	Error
Aluminum	6.11	+/- 0.16
Calcium	2.60	+/- 0.03
Carbon	3.348	+/- 0.016
Iron	4.11	+/- 0.10
Magnesium	1.20	+/- 0.02

The declared numbers on the standard reference material have a minimum number of significant digits, and that the error estimates are explicitly stated. This way of presenting the limits of uncertainty with data contains more information and is generally preferred for precision work.

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2.3: Technique C Use of Balances_1_3_1

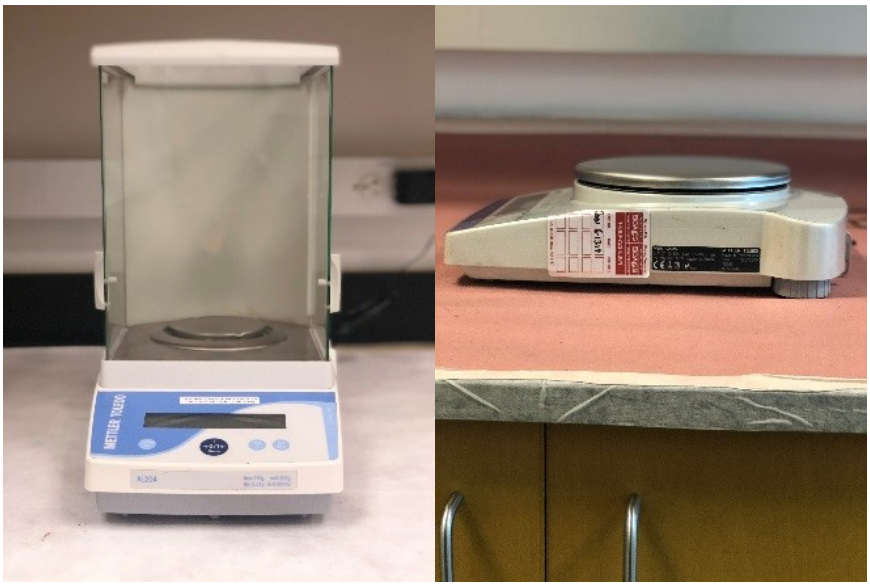
Technique C: Use of Balances

Section 1: Purpose of Technique

This technique describes how to use a laboratory balance to accurately measure the mass of a sample.

Section 2: Operations of Balances

There are generally two types of balances in the balance room. There are precision balances, which can read to the nearest 0.01 grams (g), and the analytical balances, which can read to the nearest 0.1 milligrams (mg) (0.0001 g). The visibly distinguishing factor between the two is the presence of sliding doors on the analytical balance. The precision balance in the laboratory is model PB 602-S, or equivalent. The analytical balance is model AL 204, or equivalent. They are both made by the company Mettler-Toledo, LLC of Columbus, Ohio. These balances have fundamentally identical operations.



Analytical Balance Precision Balance

Part A: Normal Weighing Procedure

1. Tare your scale before use, making sure the reading is 0. (See Part B for instructions on weighing material directly into a container).
2. Open one/two doors of the balance isolation cabinet (if using the analytical balance).
3. Place your sample on the balance pan.
4. Close side door(s) of the balance.
5. Wait until the stabilizing circle disappears.
6. Record the number as the mass of your sample.

When the number displayed no longer has a circle displayed next to it, the mass and balance has stabilized. This means weighing analysis is complete.

Some balances are sufficiently sensitive to measure water as it is evaporating from aqueous solutions, or atmospheric water as

it is hydrating the surface of reagents and equipment. In this situation, the balance may be stable and accurately displaying the correct mass, but the mass of the material you are measuring may not be constant. In this situation, select the reading that will most closely represent the true value of the material being weighed.

Note that a balance has a limit to how much weight it can read. There is normally a maximum capacity displayed on the front of the balance. **Do NOT go over this range.**

AL 204 (analytical balance)	PB 602-S (precision balance)
Max: 210 g Min: 0.01 g	Max: 610 g Min: 0.5 g

Part B: Weighing Procedure Using Tare Function

If you intend to use a container to carry your sample, you can tare (zero) the container on the balance. You will not have to subtract the initial weight of the container from the weight of the sample and the container together. However, review your procedure to see if you do need the weight of the container and be sure to record the container weight if it will be needed in the future.





1. Set the empty container on the balance pan and hit the tare button.
2. The balance should now display “0”. This allows the balance to ignore the weight of the container and accurately measure only the sample.
3. Add your sample to the container and record the weight that the balance shows.

Section 3: Maintenance of Balance Accuracy

Modern balances are marvelous devices and are precision instruments. However, any balance will not give an accurate weight if it is mishandled. Depending on balance precision, a balance may need to be adjusted to match the gravitational acceleration of its location. Moving the balance and level may affect balance measurements. Avoid moving the balance in order to preserve its reading accuracy.

If a leveling bubble is present, the feet of the balance should be adjusted until the leveling bubble is centered as shown:



Centered Leveling Bubble

Precision

The precision of a balance can be monitored by recording the weight of a stable object over time. Most commercial and government laboratories monitor balances weekly, although some places require a check prior to each use. The calibration of the balance may often be adjusted to a standard mass. Usually this adjustment is done by an independent calibration company.

Static Charge

Contact between two materials and then separating them may generate a static charge. This can introduce unknown forces (either up or down) on the balance pan and cause an error. The static can be lessened by using a balance pan with doors to insulate it. This helps with the accuracy of an analytical balance.

Thermal Effects

Weighing an object that is not at constant temperature can affect the accuracy of a balance reading. If the object is either too hot or too cold, the weight could drift due to air currents and thermal expansion of the weighing pan. A constant temperature results with increased stability and accuracy. Laboratories are generally kept at a constant temperature to prevent measurement drifting due to thermal effects.

Buoyancy Error

When weighing is carried out in air, this can create buoyancy error, which may or may not be significant. The balance is subjected to buoyancy due to being submerged in air. Taring can prevent a drift due to buoyancy to help cancel this effect, or the buoyancy of air can be subtracted by calculation. Refer to the following for more discussion and calculation:

https://chem.libretexts.org/Ancillary_Materials/Reference/Reference_Tables/Analytic_References/Appendix_09%3A_Correcting_Mass_for_the_Buoyancy_of_Air

Section 4: User's Manual for Balances

For further information on the balance in your laboratory, search online for the maker and model number. This manual can explain what features that specific balance can do which can vary between balance instruments.

https://www.labcompare.com/191234-Eliminating-Inaccuracy-in-Precision-Weighing-Caused-by-Static-Charge/

https://bitesizebio.com/33245/drift-measurements-analytical-balances/

https://www.mt.com/dam/mt_ext_files/Editorial/Generic/6/Weigh_Uncertain_Number5_0x0003d6750003db670009174c_files/measure_mass_force.pdf

https://www.mt.com/dam/product_organizations/laboratory_weighing/basic_weighing/Products/AL_and_PL/manuals/en/AL-PL-PL-S_OI_en_11780906C.pdf

https://academic.oup.com/labmed/article/35/1/48/2504342

All websites in this page were in service as of 06/26/2019.

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2.4: Technique D Vacuum Filtration_1_1_2

Technique D: Setup of Vacuum Filtration

Section 1: Purpose of Technique

Vacuum filtration is a technique for separating a solid product from a reaction mixture. The mixture is passed through filter paper in a Buchner funnel. The solid is trapped by the filter and the liquid is drawn through the funnel into the flask below. A vacuum system is used to make the filtration faster.

Only a moderate vacuum is typically needed. **Fluid is NOT to enter the vacuum system.** Care should be taken to avoid liquid getting into the vacuum system. After vacuum filtration, the solid may not be completely dry. Filtered solid material is damp or wet on the surface and might still have to be air-dried or oven-dried.

Buchner funnels are typically used for stable solids and liquids (i.e., air and water unreactive material). Special techniques for dealing with air and water reactive materials are beyond the scope of this technique.

Section 2: Components of Vacuum Filtration

Filter flask

Buchner funnel

Vacuum adapter

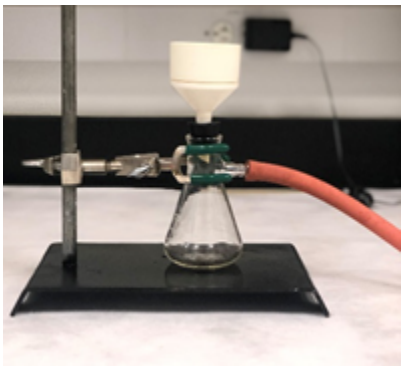
Vacuum tubing (thick tubing that resists compression)

Vacuum system (provided by the building services or a vacuum pump)

Filter paper

Stand and clamp

Set up the vacuum equipment similar to as the photo below. Your setup may not exactly look like the photo due to available equipment, but it should be functional.



Setup of Vacuum Equipment

Section 3: Setup of Vacuum Filtration

Part 1: Inspect the equipment

1. Check the equipment (filter flask, Buchner funnel, and vacuum tubing) for broken parts or cracks before use.

2. Make sure the filter paper fits in the Buchner funnel. (Folding of the filter paper is fine if it is bigger than the Buchner funnel; do not use filter paper if it is smaller than the Buchner funnel).

3. Make sure the Buchner funnel fits snugly on the filter flask.

Part 2: Putting everything together

1. Attach the filter flask to the stand using a utility clamp.

2. Place the Buchner funnel on the filter flask. Add a filter paper inside the Buchner funnel.

3. Secure the tubing to the arm of the filter flask on the side and to the vacuum system. The tubing should be long enough to attach to the vacuum located in the fume hood.

Part 3: Carrying Out the filtration

1. For each filtration, use only one filter and one filter funnel. Make sure the system is set up.

2. Turn the vacuum on slowly and gradually pour in your solution to the Buchner funnel. The solution you are filtering should consist of both a solid and liquid, not just a solid.

Section 4: Important Things to Remember

Requirements for Vacuum Filtration

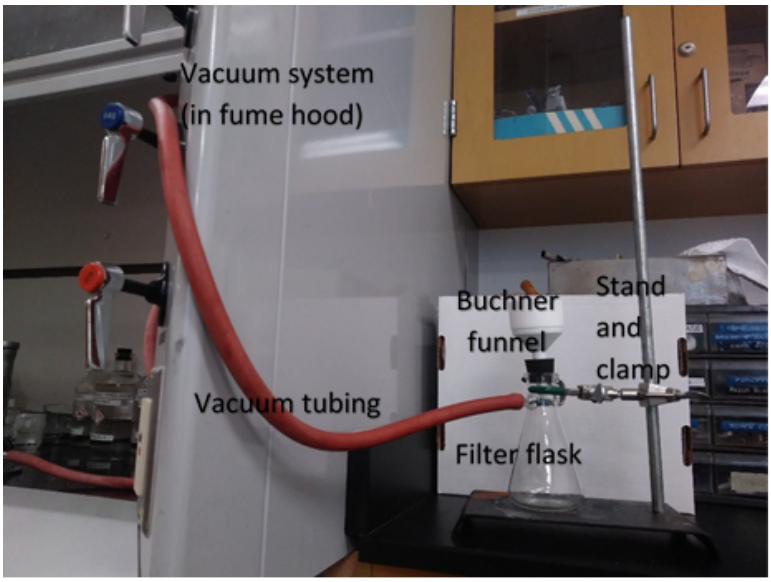
Use filter paper that is the appropriate size for the funnel. It should lie flat on the inside of the Buchner funnel and cover all of the small holes of the funnel.

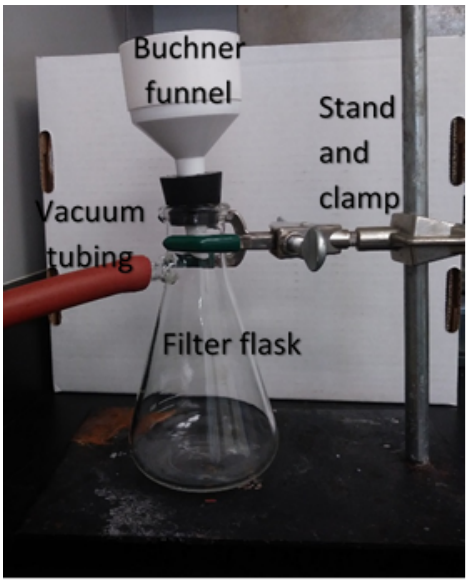
You may use deionized water to wet the filter paper enough so that it sticks to the Buchner funnel better and prevents solid substances from getting under the filter paper.



Use a utility clamp and stand to hold the filter flask down. Because you are using thick vacuum tubing for the filter flask, the tubing may cause the flask to be top-heavy. Not properly securing the filter flask can cause the filter flask to fall.

Some examples of how to set the vacuum filtration system are provided below:





When you are ready to filter, turn the vacuum on slowly and gradually pour in your solution.

In special circumstances, a liquid trap may be connected between the system and the vacuum with vacuum tubing to prevent liquid from being suctioned into the vacuum system. Remember, **fluid is NOT to enter the vacuum system.** Using this technique serves as a precaution to prevent fluid from entering the vacuum system. Use of a secondary liquid trap is unlikely needed for this class.

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2.5: Technique E Volumetric Transfer of Reagent_ 1_1_1

Technique E: Volumetric Transfer of Reagent Using Pipet

Section 1: Purpose of Technique

This technique describes how to properly use a volumetric pipet to collect, measure, and transfer a precise volume of liquid.

Section 2: Operations for Volumetric Pipet Transfer

One of the most common type of volumetric glassware is the volumetric pipet. The volumes most often used in chemistry labs are 1.00-ml (milliliter), 2.00-ml, 3.00-ml, 5.00-ml, 10.00-ml, and 25.00-ml pipets. The volumetric pipet is used to transfer a specific volume of liquid from one container to another. It is an elongated glass tube with two, slim hollow glass stems in between a glass bulb containing the marked volume of the pipet. Above the glass bulb, there is a calibration line that specifies the volume of liquid withdrawn at a specific temperature. On the bottom stem, there is a fine, tapered tip that allows a precise stream of liquid to be dispensed. A rubber pipet bulb is used to generate suction and control the amount of reagent collected by the volumetric pipet.

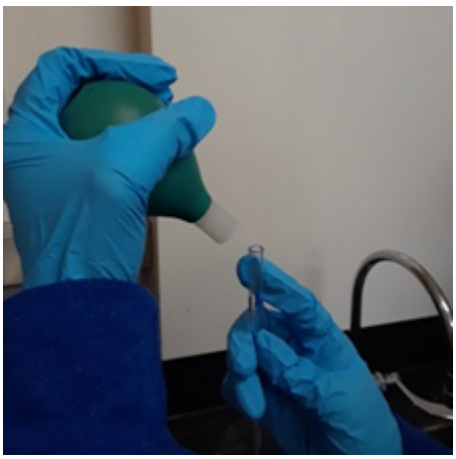
1. Obtain two clean and dry beakers or other containers. One will be used for stock reagent. One will be used to receive the dispensed liquid.

2. Collect enough reagent for procedure. Obtain a volumetric pipet of the appropriate volume for the experiment and a rubber pipet bulb.

Notes:

- Either start with a dry pipet, or the pipet will need to be rinsed with a little of the solution that you are measuring.
- Several styles of pipet bulbs are available. The style shown does not affix to the top of the pipet, it is only pressed briefly at the end of the pipet to generate a temporary seal.

3. Hold the pipet from the top stem, expel air from the pipet bulb. Hold the pipet bulb on the top of the pipet and submerge the pipet tip below the surface of the reagent. Slowly release the pipet bulb to provide suction and fill above the calibration line of the pipet.





4. Stop suctioning when the meniscus is a few centimeters above the calibration mark. Keep the pipet tip submerged in the solution you are measuring. Be careful as the height of the liquid being sucked into the pipet gets faster as you get to the top. Align your eye to the calibration mark so that is appears as a single paint line.

5. When the liquid is above the calibration mark, remove the pipet bulb and immediately cover the opening of the pipet with your index finger. Gently release the seal with your finger to allow the solution to drain from the pipet due to gravity. Let the meniscus move down until it is just touching the paint line of the calibration mark.

Incorrect
- Meniscus is not aligned with the calibration line.



Correct
- Meniscus is right on the calibration line.



6. Withdraw the pipet from the solution and quickly wipe the exterior of the pipet to make sure there are no remaining liquid droplets. Place the pipet over your receiving container and release your index finger to allow the liquid to stream down. Once the pipet has fully drained, wait an addition 15 seconds to ensure that all the solution had been delivered.

7. Tap the surface of the liquid three times with the tip of the pipet to reduce any surface tension built up in the beaker. Do not blow out the last drop from the pipet. The transfer pipet is calibrated to contain this last drop.

Section 3: Tips for a Precise Measurement

The balance will not give a precise measurement of volume if the volumetric pipet is mishandled or not cared for. When handling a volumetric pipet, there are certain factors that should be evaluated before throughout the procedure:

1. The tip of the pipet should always be examined before the experiment begins to check if there is any damage that can affect the accuracy of the measurement. If the tip is chipped or shows any sign of damage that contacts the center hole of the pipet tip, insufficient surface tension will be present to maintain a constant size of the remaining drop in the transfer pipet. The pipet is likely not repairable and should be discarded.

2. It is highly important to hold the pipet from the top stem rather than in the middle. The middle of the pipet is made of very thin glass and can easily be broken when lateral force and torque is applied.

3. Before placing the pipet bulb onto the opening of the pipet, squeeze the bulb to expel air before contacting it on top of the pipet. This will seal any air around the pipette that will make it hard to collect any liquid. It is not necessary to squeeze the bulb so vigorously to capture liquid. Slow presses on the bulb will allow enough liquid to come up and will give you control on the speed the liquid is collected at.

4. If the meniscus is not a little higher than the calibration mark while the pipet is submerged into the liquid, then the volume collected will not result in a precise mass. Once the pipet is taken out of the liquid, the change in volume allows the liquid to decrease which reduces the amount of mass the balance will read.

5. When transferring liquid using the volumetric pipet, it is important to use your index finger to stop the flow of liquid rather than your thumb. Thumbs tend to have larger areas of contact, which lead to less control of releasing pressure.

6. While the reagent is streaming down, do not let the tip of the pipet hit the liquid's surface or the sides of the beaker.

Section 4: Further Information

Getting accurate results using the volumetric pipet takes practice. In order to achieve precise measurements of volume for an experiment, you must know how the pipet works and how to take care of it properly. For further information on using a volumetric pipet, check out the resources provided below.

https://chem.libretexts.org/Ancillary_Materials/Demos%2C_Techniques%2C_and_Experiments/General_Lab_Techniques/Use_of_a_Volumetric_Pipet

http://faculty.sites.uci.edu/chem1l/files/2013/11/RDGvolflaskpipet.pdf

http://dept.harpercollege.edu/chemistry/chm/100/dgodambe/thedisk/labtech/piptech.htm

http://www.ehs.pitt.edu/workplace/pipetting.html

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2.6: Technique F Use of Crucible _1_1_1

Technique F: Use of Crucible

Section 1: Purpose of Technique

This technique describes how to use a laboratory crucible to safely heat a sample.

Section 2: Operations of Crucible

There are several types of crucibles that can be used for heating up materials. Possible types of crucibles are alumina, ceramic and porcelain. Most labs require the use of porcelain crucibles. Porcelain crucibles are glazed on the outside and inside, except there is usually no glaze on the bottom. Crucibles are meant to withstand high temperatures of 1020-1050oC. Crucibles can heat up a variety of materials like metals (cooper, cobalt, etc.), hydrates and glass.

1. Use crucible tongs to pick-up crucibles and prevent fingerprints.
2. Put crucible on a clay triangle that has been placed over an iron ring attached to a stand and placed above a Bunsen burner.
3. Make sure that the crucible is directly in the center of clay triangle to prevent it from falling over.
4. After the crucible is in place, turn on the Bunsen burner flame (**CAUTION:** DO NOT use immediate high heat on the crucible) and begin heating.

Section 3: Maintenance of Crucible

The proper way to prepare the crucible and lid is to wash it with laboratory water **ONLY.** Use a gentle flame and heat the crucible for two minutes or until water is evaporated. This will allow the crucible time to be completely water free prior to more intense heating.

Place the clean crucible and lid on the wire gauze. **Do not** place the clean crucible or lid on the countertop. Clean it again if it is placed on the countertop.

Section 4: Safety Precautions of Crucible

- **DO NOT USE** broken or severely damaged crucibles. Thermal shock can break the crucibles if there are already defects in the crucible.
- **DO NOT** overheat the crucible. It may cause materials to spatter or to adhere the bottom
- **USE ONLY** crucible tongs to put on the lid when the crucible is over a flame.
- **DO NOT** touch a hot crucible with your hands. It can burn your hands.
- **NEVER** put down a hot crucible has been heated on the counter. **USE** a wire gauze.
- **NEVER** directly pick-up the crucible after it has been heated up. Allow the crucible to cool down first before using crucible tongs.
- **DO NOT** stand near the crucible, especially when material is heating up. Make sure to use the lid before turning up the flame on the material, as you **DO NOT** want the material to become ejected toward you.
- **DO NOT** allow cold water to interact with the hot water inside the crucible, it can cause thermal shock.
- **DO NOT** allow cold water to touch the crucible after it has been heated up. It can cause mechanical shock of the crucible.

Do's

Do use crucible tongs to pick up and put down the crucible and lid on the wire rack.



Do use crucible tongs to place the lid on the heated crucible.

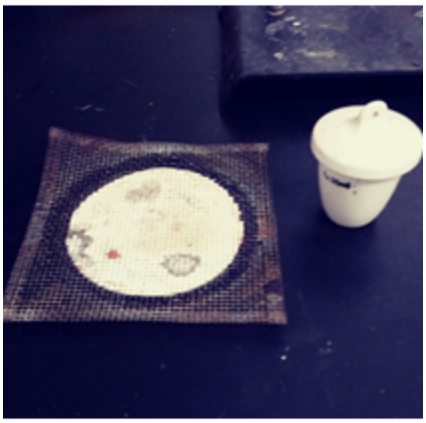


Do place the lid and crucible after cleaning onto the wire gauze **and** when the crucible and lid are hot.



Do not's

Do not place the crucible and the lid on the counter at **ANYTIME** during the cleaning and experiment.



Do not place the crucible on its side. Make sure it is centered on the clay triangle.



https://www.indianscientificinstrument.com/laboratory-porcelainware/porcelain-crucible.html

http://www.morganmms.com/media/1766/crucliblecareuseandsafetyinformation-mgam.pdf

https://chem.libretexts.org/Ancillary_Materials/Demos%2C_Techniques%2C_and_Experiments/General_Lab_Techniques/Heating_a_Crucible_to_Constant_Weight

https://chem.libretexts.org/Ancillary_Materials/Demos%2C_Techniques%2C_and_Experiments/General_Lab_Techniques/Heating_a_Crucible_to_Constant_Weight

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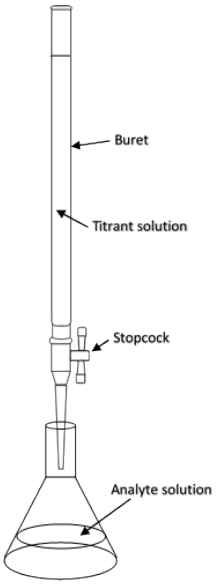
2.7: Technique G Buret Use_1_1_2

Technique G: Buret Use

Section 1: Purpose of Technique

A buret is primarily used for titration to determine the concentration of an unknown solution by adding a solution of known concentration. Titration is a type of quantitative analysis and the most common forms are acid-base, precipitation, complexometric, and redox titrations. Because the purpose of titration is to find the concentration of an unknown solution, it is very important that accurate measurements are taken to determine the volume of the known solution added to bring a reaction to an endpoint.

The *titrant* is the solution with known concentration and is placed in the buret. The *analyte* is the solution of unknown concentration that is being measured. The analyte is usually placed in an Erlenmeyer flask with an indicator. The equivalence point is the exact point where the moles of the titrant solution and the analyte solution are equivalent. The end point is when the indicator in the solution shows a color change. This usually occurs close to and just after the equivalence point is reached.



Using a buret as opposed to other measurable glassware, such as pipets and graduated cylinders, makes it easier to control the delivery of and measure the precise amount of solution being delivered. Slight turns of the stopcock located on the buret can control the flow of solution and there are methods to get as close as possible to the end point without going over. Because of its smaller meniscus, a buret is more accurate and precise than a graduated cylinder. A measurement made with a buret should be to the hundredths place (ex: 0.00). The burets generally used in the laboratory titration procedures hold up to 50 millimeters (ml). 10 ml, 25 ml and 1.0 ml burets are also commercially available.

Section 2: Components for Using a Buret

Buret

Stand

Buret clamp

Deionized water bottle

Erlenmeyer flask

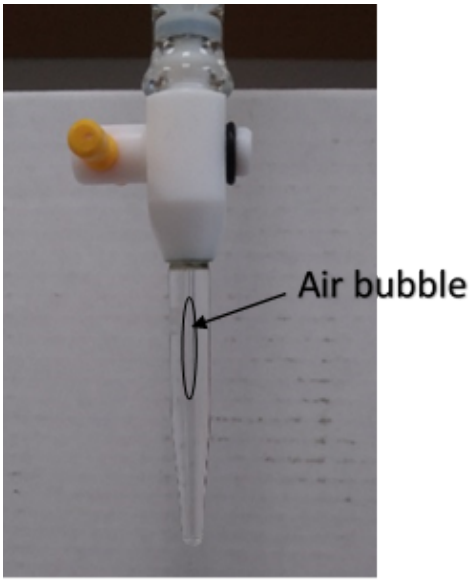
Waste beaker

Section 3: Setup of a Buret

Part 1: Inspect the equipment

1. Check the equipment for broken parts before use.
2. Inspect the glassware and the tip of the buret. As long as the bore of the buret tip is intact, the buret is functional.

Part 2: Preparing the Buret

1. Before titration, the buret must be rinsed with laboratory water and with the solution you are titrating with. Rinse the buret with small volumes laboratory water three times. Then, rinse the buret with the titrant solution three times, making sure to let the solution coat the inside of the buret completely and run through the tip.
2. 

Check for air bubbles in the tip of the buret. Air bubbles trapped in the tip can throw off your measurements. If you see an air bubble, you can get rid of it by filling the buret with a solution, firmly holding it over a sink, and vertically shaking it up and down in one quick motion.

Part 3: Setting up the Buret Apparatus

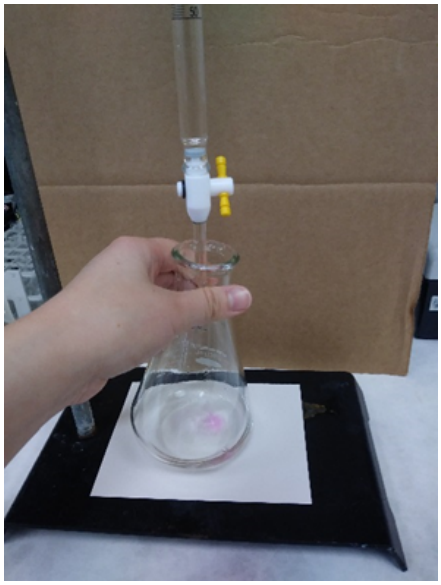
1. After the buret has been rinsed with the laboratory water and the solution, fill the buret with the solution, going above the 0.00 graduation mark.
2. Attach the buret to a stand using the buret clamp.
3. Place the waste beaker under the tip of the buret. Stand at eye level to the 0.00 graduation mark and turn the stopcock slightly to release some of the solution into the waste beaker. Try to get the meniscus to reach the 0.00 line as your initial measurement. It is okay if you go past the 0.00 line. Just make sure to record the initial measurement accurately for what it is.

Part 4: Performing the Titration

1. Fill the Erlenmeyer flask with the required amount of the analyte solution and make sure to **add indicator**. Adding indicator is the only way to visibly see whether you reached the end point or not. Place the Erlenmeyer flask under the tip of the buret.
2. Turn the stopcock of the buret, releasing the titrant solution into the Erlenmeyer flask. Control the flow of the titrant by adjusting the stopcock.
3. Observe the analyte solution for changes in color from the indicator. Swirl the solution in the flask to make sure the titrant is evenly distributed. You know when you are close to the end point if the small bursts of color in the flask persist for more than a few seconds. Slow down the release of titrant into the flask by turning the stopcock as you near this point. Begin adding half drops instead of full drops if necessary. Titration is complete once the analyte solution reaches a uniform color. The end point is reached when the indicator color is faint. A more vibrant color may indicate that the end point was exceeded. If the

endpoint was significantly surpassed, you will have to redo the titration.

The following photos show the general setup and a faint pink for the endpoint (phenolphthalein is the indicator used in the photos—other indicators may be used in the titration).

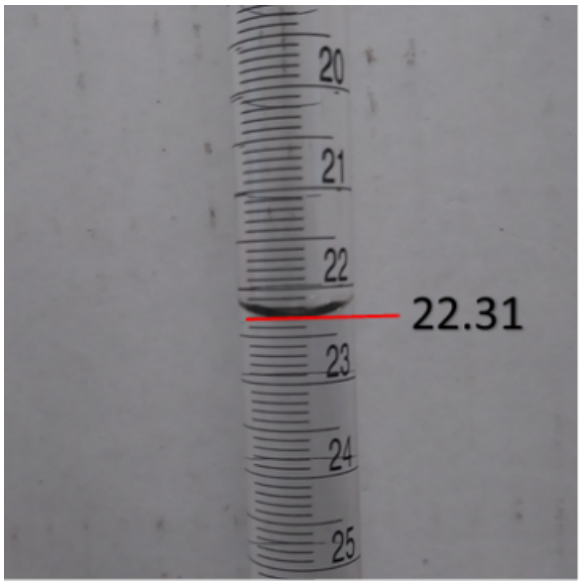




Section 4: Important Things to Remember

Measure your buret to the nearest 0.01 ml

When taking measurements using the buret, make sure to view the meniscus at eye level. You should be looking straight at the line where the meniscus lies. The graduation line when looking directly at it should not form an oval.





In the left photo above, note that the inscribed circle at the 1.00 ml mark looks like an oval. In the right photo above, note the inscribed circle at the 20.00 ml mark looks like an oval, the 22.00 ml mark looks like a line, and the 24 ml mark again looks like an oval. If the mark closest to the meniscus is a line, it means that your eye is at the correct height to properly read the buret.

People are not generally good at estimating tenths, but are good at splitting by halves. Use this technique to estimate the final 0.01 ml reading between the tenths of a milliliter mark. Mentally draw a line ½ way between the two marks. This will be the 0.05 ml between the two tenth milliliter marks. Repeat this process to split the distance between the 0.05 ml mark and the inscribed

mark. This will be a 0.025 ml increment. Evaluate where the meniscus is now relative to your estimates and round either up or down to the final reading.

For example, if your meniscus is between 22.30 ml and 22.40 ml, then your buret should be evaluated like this:

22.30 _____

22.325 - - - - - - - - - - - -

22.35 ----------

22.375 - - - - - - - - - - - -

22.40 _____

Now you should be able to accurately estimate where the meniscus falls on the buret to the nearest 0.01 ml.

Use your information

If you are doing multiple titrations, there is nothing wrong with doing calculations to estimate the volumes for your second, third, and fourth titrations. Recognize that your calculation is just an estimate and may not reflect the actual results of the titration procedure. The only way to know the actual concentration of the analyte is by doing the titration multiple times.

For example: You are performing an acid-base titration between hydrochloric acid (HCl) and sodium hydroxide (NaOH). The concentration of the sodium hydroxide is 2.1043 M and you are trying to determine the concentration of 15.00 ml of HCl. After your first titration, your final buret measurement is at 17.35 ml. Use this calculated estimate as a guide for the next titrations.

$HCl + NaOH \rightarrow H_2O + NaCl$

$$M_{\mathrm{HCl}} = \frac{M_{\mathrm{NaOH}} V_{\mathrm{NaOH}}}{V_{\mathrm{HCl}}}$$

$$M_{HCl} = \frac{(2.1043 M \mathrm{NaOH})(17.35\ \mathrm{mLNaOH})}{15.00\ \mathrm{mLHCl}}$$

$$M_{HCl} = 2.434 M \mathrm{HCl}$$

For your second titration, you decide to use a 20.00 ml volume of HCl instead of a 15.00 ml volume. Using a little algebra, the amount of 2.1043 M NaOH that is expected to be added is given by:

$$V_{\mathrm{NaOH}} = \frac{M_{\mathrm{HCl}} V_{\mathrm{HCl}}}{M_{\mathrm{NaOH}}}$$

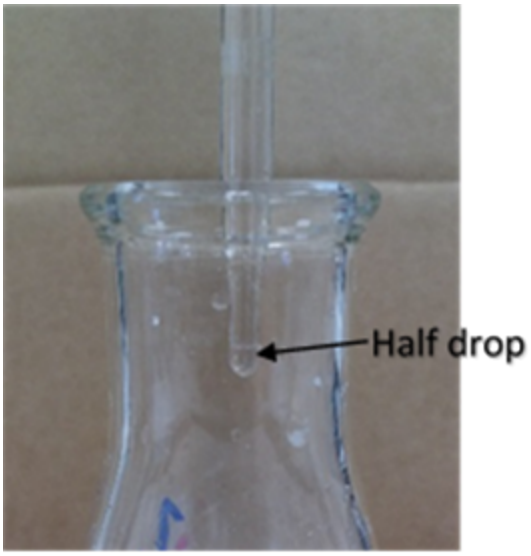
For our example: $V_{\mathrm{NaOH}} = \frac{2.434 \mathrm{MHCl} \times 20.00 \mathrm{mlHCl\ solution}}{2.1043 \mathrm{MNaOH}}$

For our example: $V_{\mathrm{NaOH}} = 23.13 \mathrm{ml}$ NaOH titrant (estimated)

Therefore, for your second titration, dispense down about 20-22 ml of titrant before carefully adding the last drop to the endpoint.

Adding titrant by half-drops

If you think you are nearing the end point, instead of allowing the buret to release full drops one at a time, you can create half-drops. To do this, close the stopcock until the buret is no longer releasing the titrant. Carefully open the stopcock slightly so that a drop forms at the tip of the buret, but not enough to fall. Turn the stopcock off. Use a deionized water bottle, and wash the tip of the buret so that the drop of titrant falls into the titration. This is considered a half-drop. Remaining fluid on the tip of the buret at this point should be entirely laboratory water. The next full drop that the buret releases from the tip will actually be another half drop of titrant (combined with the remaining water on the tip). You can alternate back and forth to add half-drop quantities of titrant.





First titration quick method

Often, you have no idea where the first titration endpoint occurs. This makes it quite easy to accidentally exceed the endpoint and add too much titrant, with resulting accuracy error. One technique to work around this is to reserve a little bit of analyte solution. Pour a small volume of analyte into out into a clean, small beaker. It is considered reserve because if you accidentally over shoot the end point, you can use reserve to save your analysis. The titrant can be added relatively quickly until the indicator changes color.

The following photos show how this works. A small amount of analyte is temporarily transferred to a small beaker. The titration is rapidly done dropwise until the indicator changes. The reserve is returned to the Erlenmeyer beaker to change the indicator back, and you now know you are close to the endpoint.







Now, use the half-drop technique described earlier to finish the titration. Remember to return the reserve to the Erlenmeyer flask regardless if it were needed. After you have completed one titration, subsequent volumes can be estimated as was described above.

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2.8: Technique H Use of a Bunsen Burner_1_1_1

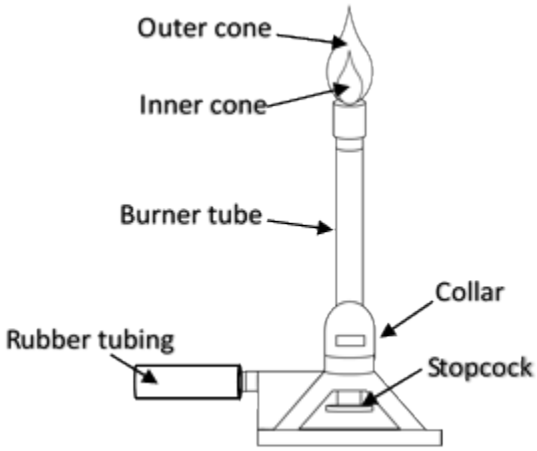
Technique H: Use of a Bunsen Burner

Section 1: Purpose of Technique

A Bunsen burner is a piece of equipment used for the general purpose of heating substances. When connected to a gas line and lit with either a striker or a match, Bunsen burners generate a controllable, open flame that is hot enough for most classroom experimental procedures. The flame produced consists of a visible outer cone and inner cone, in which the topmost tip of the inner cone is the hottest part of the flame (See Figure 1).

In case of emergency, completely shutting off the gas line is the best way to stop the flame. Make sure the gas line is completely off at all times unless the Bunsen burner is in use.

The maker of the Bunsen burners is Humboldt and the model is PN 6200.1. These are the standard Bunsen burners used for classroom laboratory experiments but other models and brands exist. Certain attachments are available for Bunsen burners such as fan adaptors (not shown) that can change the shape of the flame as desired.



Section 2: Equipment needed for a Bunsen Burner

Bunsen burner

Gas source

Rubber tubing (gas tubing)

Striker or matches

Section 3: Setup

Part 1: Inspect the equipment

Figure 1

1. Check the equipment for broken parts before use.
2. Inspect the rubber gas tubing for cracks by stretching it and looking for any major cracks throughout the tubing. If the tubing has cracks and leaks, discard it and get a new tubing.

Part 2: Assemble everything together

1. Attach one end of the rubber tubing to the arm of the Bunsen burner and the other end to the gas line located on your lab bench.

2. Get a striker or some matches.

Part 3: Lighting the Bunsen burner

1. Once the Bunsen burner and rubber tubing is secure, turn on the gas line.

2. If using a striker, squeeze the handle of the striker over the top of the Bunsen burner to generate a spark where gas is emitted. Do this until a flame appears. Adjust the size of the flame using the gas line knob or the stopcock as needed.

3. If using matches, strike the head of the match against the striking strip on the back of the pack. Hold the flame up to the top of the Bunsen burner where gas is emitted until a flame appears. Adjust the size of the flame using the gas line knob or the stopcock as needed.

Section 4: Important Things to Remember

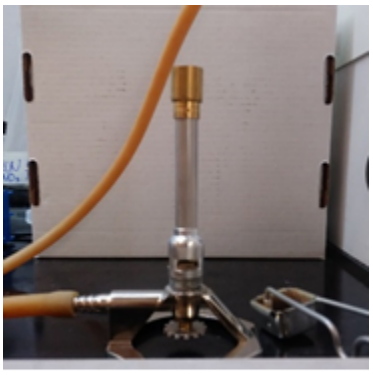
Precautions and tips for using a Bunsen burner

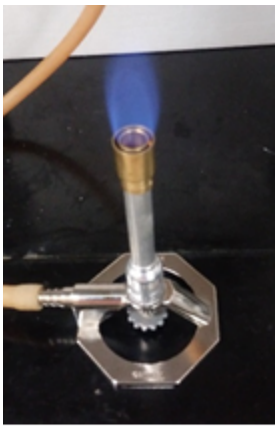
The gas lines located in the lab benches and in the fume hood have a gas pressure of 4 pounds per square inch (psi). For reference, keep in mind that the pressure needed to blow up a balloon is 15 psi (atmospheric pressure). The pressure of the gas lines is not a dangerous amount, but always be aware of its use. When the gas line is on, you will normally hear a slight hissing sound. Natural gas has no smell, but a trace amount of a sulfur compound is added so that it does have odor. You might begin to smell it from unburned gas. To avoid leaks, you should not have the Bunsen burner or the gas line on when not in use.

Bunsen burners are generally safe to use as long as they are used properly and with common sense. For example, the Bunsen burner should be kept away from the edge of the table or anywhere it could easily be knocked over. Do **not** reach over the Bunsen burner, even if it is off. Be cautious when heating glass containers. If the container is too cold (from either a reaction, ice bath, or other freezing procedure), applying direct heat to the glass may cause it to crack or break.

There are several features on the Bunsen burner meant for controlling the size of the flame, but generally, the most effective way is by controlling gas valve located on your lab bench or in the fume hood. The gas valve controls the amount of gas entering the Bunsen burner. Allowing more gas to enter the Bunsen burner creates a larger flame. Twisting the collar clockwise will reduce the amount of air entering the Bunsen burner, cutting off oxygen supply to the flame. The flame can also be adjusted by using the stopcock located at the bottom of the Bunsen burner, which adjusts the flow of gas entering the Bunsen burner.

Here are some pictures of a Bunsen burner set up:




The hottest part of the flame is located at the tip of the inner blue cone.

Additional safety tips

Never leave an unattended flame.

Check the area around your Bunsen burner for flammable objects (paper towels, flammable solvents, etc.) prior to lighting the flame.

Hot objects look just like cold objects. Wait sufficient time for objects to cool before handling them.

Metals can conduct heat very well. Be aware that this may happen when heating objects with clamps.

Loose hair and clothing should be tied down to avoid contact with any flame.

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2.9: Technique I Use of Spectrophotometer_1_1_1

Technique I: Use of Spectrophotometer

Section 1: Purpose of Technique

This technique describes how to use a laboratory spectrophotometer.

Section 2: Operations of Spectronic 200E

There are several types of spectrophotometers that are used to determine the amount of light absorbed within a given sample solution. The spectrophotometers used in the lab are the Spectronic 20, Spectronic 20+ and Spectronic 200. Before using the Spectronic 200, follow these steps to make sure that no damage or injury will occur due to faulty or careless behavior.

NOTE: Several terms are used for the container used to hold a sample in a spectrometer. This procedure will use the term 'optical cell' but other terms like 'cuvette' may be used in reference texts and instrument manuals.

1. Make sure the spectrophotometer is on a stable, flat surface free from vibrations.
2. Plug in the spectrophotometer and turn it on by flipping the power switch at the back of the machine. The following image will appear on the screen when it is on. (Figure 1)



Figure 1. On screen

3. Make sure that the sample compartment is empty. Then, press ENTER. The following image will appear on the screen. (Figure 2)



Figure 2

4. Look at the image below. Once at this point, wait for approximately 1 minute.

5. The screen will then display the Home Menu. Use the arrows to select the SPEC 200E MODERN INTERFACE option, then press ENTER. (Figure 3)



Figure 3. SPEC 200E MODERN INTERFACE

6. The screen will then show APPLICATION LIVE DISPLAY. Press the right arrow. (Figure 4)



Figure 4. APPLICATION LIVE DISPLAY

7. The screen will then show APPLICATION MULTI λ, press the right arrow again. (Figure 5)

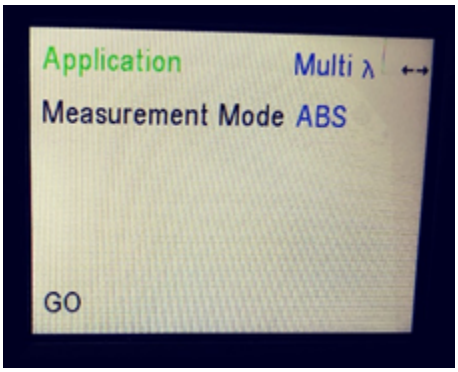


Figure 5. APPLICATION MULTI λ

8. Then the screen will show APPLICATION QUANT, press the right arrow again. Refer to figure 6.

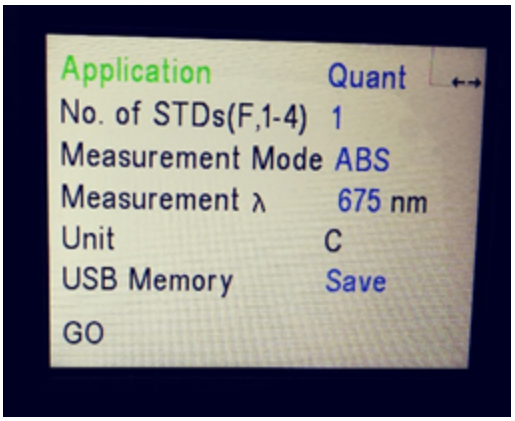


Figure 6. APPLICATION QUANT

9. Then the screen will show APPLICATION ANALYZER, press the right arrow again. Refer to figure 7.

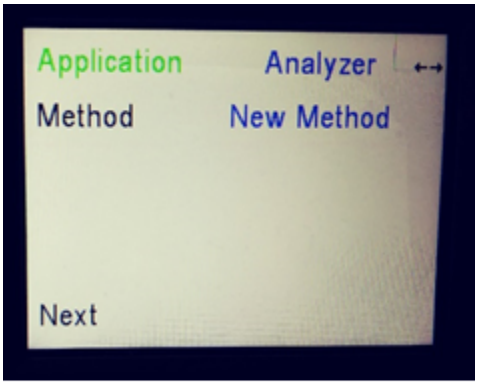


Figure 7. APPLICATION ANALYZER

10. Then the screen will show APPLICATION SCAN, press ENTER. Refer to figure 8.

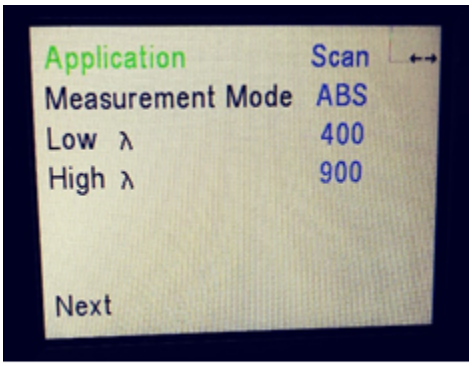


Figure 8. APPLICATION SCAN

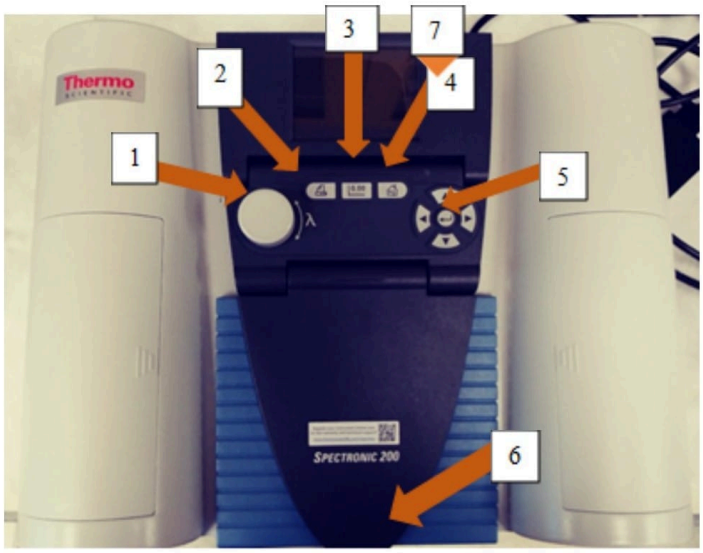
11. Fill an optical cell half-way with laboratory water. Use Light-Duty Tissue Wipers, to wipe off any fingerprints on the outside of the cell.

12. Place the optical cell into the sample compartment, then press the AUTOZERO KEY.

13. After the spectrophotometer is done zeroing, replace the optical cell filled with water with an optical cell filled with your chemical sample.

14. Wipe the optical cell with the Light-Duty Tissue Wipers and place it into the sample compartment. Press ENTER.
15. After the spectrophotometer is done analyzing the sample, a graph will appear to the right of the screen.
16. Use the right arrow to move cursor to the peak of the curve. This will show the peak absorption.
17. Press the HOME KEY when you are done. To turn the spectrophotometer off, flip the power switch on the back of the machine.
18. **DO NOT** move the spectrophotometer if it is plugged in, as it may cause damage to it. **DO NOT** yank the cord out of the wall, but gently pull from the base of the plug on the cord.
19. If there are any issues with the use of Spectronic 200, refer to the link below:

https://archive-resources.coleparmer.com/Manual_pdfs/02652-55.pdf



1. Wavelength Knob
2. Printer Key
3. Autozero Key
4. Home Key
5. Enter
6. Sample Compartment
7. Power Switch

Figure 9. Spectronic 200

Section 3: How to Use the Spectronic 20 and 20+

To minimize any potential problems, follow these steps:

1. Turn on the spectrophotometer by turning the power switch located on the left side of the instrument. Allow for the instrument to warm up for 15 minutes.
2. After warming the machine up, use the Wavelength Control Knob to set to the wavelength that you want.
3. Adjust the filter lever to the needed position for the wavelength being used.
4. Set the mode to 100% T (Transmittance) or 0 A (Absorbance) by turning the knob under the wavelength knob. This may take several turns.
5. Fill a clean Spectronic 20 tube with laboratory water as a blank solution and wipe it with a KIMwipe™ to remove liquid droplets, dust, and fingerprint residue.
6. Place the Spectronic 20 tube in the sample compartment and align it with the guide mark within the compartment. Ensure that the optical cell is firmly placed in the compartment before closing the compartment cover.
7. Make sure that the absorbance of the water sample is at 0 by using the 100% T or 0A knob to adjust the absorbance accordingly.

8. Remove the tube from the sample compartment and discard the water.
9. Rinse the tube twice with whatever solution is being analyzed and then fill it half way with the solution.
10. Repeat steps 5 and 6, then read the screen to determine how much light is being absorbed by the solution (Absorbance).
11. For an accurate reading, make sure that the needle aligns with its reflection in the mirror.
12. Remove the Spectronic 20 tube from the sample compartment then close the lid of the compartment.
13. Turn off Spectronic 20 by turning the 100% T knob counterclockwise until it clicks.
14. If there are any issues with the use of Spectronic 20 and 20+, refer to the link below: http://fg.cns.utexas.edu/fg/course_notebook_appendix_ii_files/Spectronic_20_D.pdf

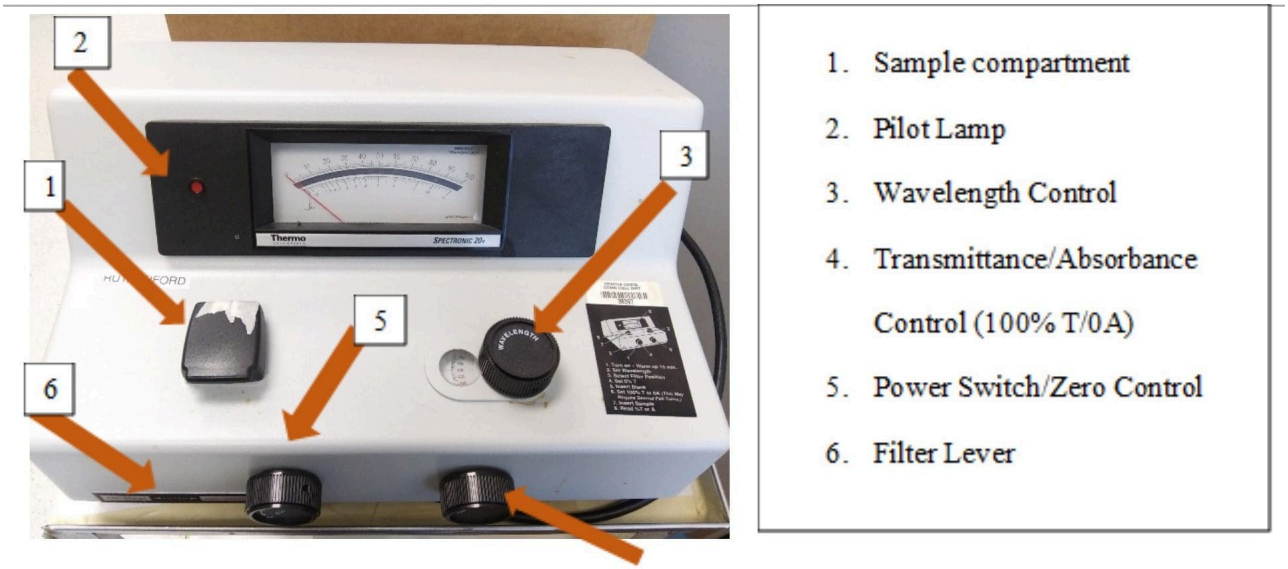


Figure 10. Spectronic 20+

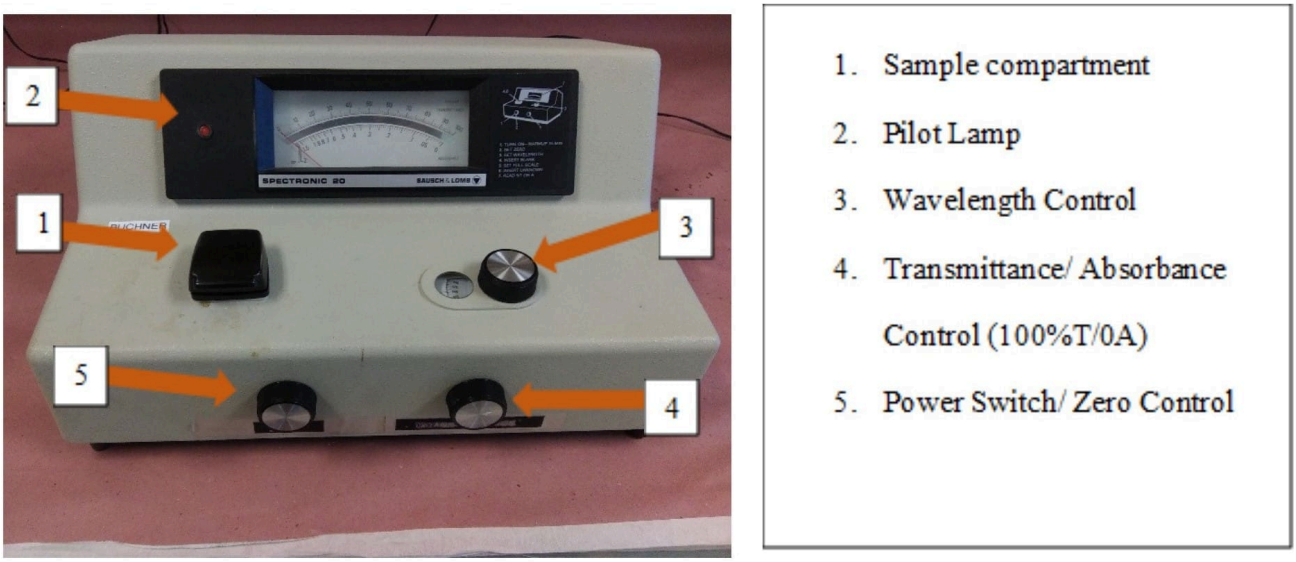


Figure 11. Spectronic 20

Section 4: Tips for Using Spectrometers

To minimize any potential problems, follow these steps:

1. Do not touch the bottom of the optical cell (or tube) when placing it in the spectrophotometer. To ensure that optical cell (or tube) is clean, use a paper towel or Light-Duty Tissue wipers to wipe away fingerprints.
2. Make sure to fill the optical cell (or tube) half way with the solution to ensure that all of the light path in the spectrophotometer is used.
3. When pouring the solution into the optical cell (or tube), make sure there are no bubbles present in the solution as this could causes potential errors.
4. For Spectronic 20+ make sure to align the solution to the mark in the front of the instrument.

Section 5: References

https://chem.libretexts.org/Bookshelves/Physical_and_Theoretical_Chemistry_Textbook_Maps/Supplemental_Modules_(Physical_and_Theoretical_Chemistry)/Kinetics/Reaction_Rates/Experimental_Determination_of_Kinetcs/Spectrophotometry

http://fg.cns.utexas.edu/fg/course_notebook_appendix_ii_files/Spectronic_20_D.pdf

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Sample Word 1 | Sample Definition 1

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