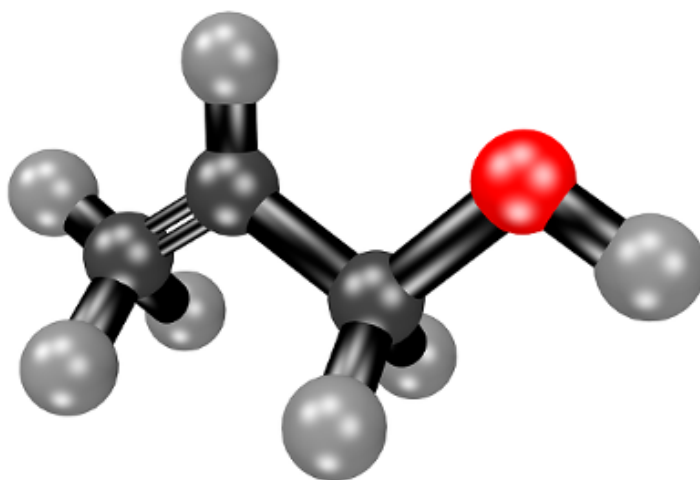# Experiments in College Chemistry

AN OPENTEXTBOOK
Version 2.8 - Fall 2023

Science Department
B.M.C.C.
The City University of New York

THIS BOOK NEEDS TO BE SPIRAL BOUNDED + PRINTED DOUBLESIDED

Contents

1 Safety

1. Flammable materials, like alcohol, should never be dispensed or used near. Chose the most correct answer.

    (a) an open door.

    (b) an open flame.

    (c) another student.

    (d) a sink.

2. If a laboratory fire erupts, immediately

    (a) notify your instructor.

    (b) run for the fire extinguisher.

    (c) throw water on the fire.

    (d) open the windows.

3. Approved eye protection devices (such as goggles) are worn in the laboratory

    (a) to avoid eye strain.

    (b) to improve your vision.

    (c) only if you do not have corrective glasses.

    (d) any time chemicals, heat, or glassware are used.

4. If you wear contact lenses in the school laboratory,

    (a) take them out before starting the lab.

    (b) you do not have to wear protective goggles.

    (c) advise your science instructor that you wear contact lenses.

    (d) keep the information to yourself.

5. After completing an experiment, all chemical waste should be

    (a) left at your lab station for the next class.

    (b) disposed of according to your instructor's directions.

    (c) dumped in the sink.

    (d) taken home.

6. You are heating a substance in a test tube. Always point the open end of the tube

    (a) toward yourself.

    (b) toward your lab partner.

    (c) toward another classmate.

    (d) away from all people.

7. Personal eyeglasses provide as much protection as

    (a) a face shield.

    (b) safety glasses.

    (c) splash proof chemical goggles.

    (d) none of the above.

8. Long hair in the laboratory must be

    (a) cut short.

    (b) held away from the experiment with one hand.

    (c) always neatly groomed.

    (d) tied back or kept entirely out of the way with a hair band, hairpins, or other confining device.

9. In a laboratory, the following should not be worn.

    (a) Loose clothing.

    (b) Dangling jewelry.

    (c) Sandals.

    (d) All of the above.

10. If acid is splashed on your skin, wash at once with

    (a) soap.

    (b) oil.

    (c) a weak base.

    (d) plenty of water.

11. Draw a diagram of your science room and label the locations of the following:
    ❏ Fire extinguisher(s)
    ❏ Exits
    ❏ Eyewash station
    ❏ Emergency shower
    ❏ Closest fire alarm station
    ❏ Waste disposal container(s)

12. Store your books and bags:

    (a) on your lab bench.

    (b) in the aisle between lab benches.

    (c) in a cabinet in the lab.

    (d) under your bench.

    (e) on the top of the instructor's desk.

13. You are excused from wearing goggles

    (a) if you have permission from your doctor.

    (b) if they are uncomfortable to wear.

    (c) during the lecture in the lab.

    (d) if you are finished with the experiment.

    (e) None of the above.

14. If you do not wear goggles during an experiment

    (a) the instructor will suggest you wear them.

    (b) your instructor will not care.

(c) you are not required to wear goggles.

(d) you will invited to leave the lab for 15 mins.

(e) None of the above.

15. Drinks and food in the lab?

(a) Are always allowed.

(b) Never allowed.

(c) Are allowed if you are hungry or thirsty.

(d) Are allowed if you are on a diet.

(e) None of the above.

16. If you feel ill in the lab

(a) call your family.

(b) ask your coworker to help you do the experiment.

(c) tell the instructor.

(d) leave immediately.

(e) none of the above.

2   Lecture policies

<div style="border:1px solid">

Lecture policies

1. You need to bring a calculator to class and your phone does not count as one.

2. There are no make-ups for missed quizzes.

3. Class work can only be done during class, hence your attendance is critical for this component of the grade.

4. If you miss a class you miss the grade of the corresponding class work carried during that class.

</div>

3   Lab policies

<div style="border:1px solid black">

<p align="center" style="color:red">Lab policies</p>

1. You will not be allowed to carry any work in the lab unless you submit all pages of the lab release form via BB.

2. You need to bring a calculator to class and your phone does not count as one.

3. Pre-lab should be carried out at home and presented to the instructor at the beginning of the experiment. An empty pre-lab page will be collected with your name in case the work was not done.

4. After the instructor evaluates and signs your work, results will be submitted via BB as a single pdf on the same day. Late submissions will be penalized with 5 points a day.

5. There are no make-ups for missed lab sessions. However, I will allow a single absence without penalty.

6. You will not be allowed in the lab unless you bring your safety goggles. Safety glasses will not be permitted

7. You will not be allowed in class unless you bring either a printed copy of the lab with all pages stapled or a bound book with all lab experiments. Loose sheets will not be accepted as are unprofessional. E-books are not allowed.

8. If you miss a lab session you miss the grade of the corresponding work carried out during that session.

</div>

4 Policies quizz

1. You should arrive to the lab

    (a) always late.

    (b) always on time.

    (c) within the first 20 mins.

    (d) None of the above.

2. Thinking about missing a lab session?

    (a) You are not allowed to miss any lab sessions.

    (b) You are allowed to miss a single lab session.

    (c) You can miss two labs if its justified.

    (d) None of the above.

3. When you are done with the experiment

    (a) you should stay until the end of the session.

    (b) you are allowed to leave.

    (c) you should help your coworkers.

    (d) none of the above.

4. The lab counts for the final grade?

    (a) Yes. It is 10% of the grade.

    (b) Yes. It is 25% of the grade.

    (c) Nope, it does not count for your grade.

    (d) None of the above.

5. Regarding the lab manual,

    (a) you should not bring it to each lab session.

    (b) you can bring loose sheets to the lab.

    (c) you can bring an e-book.

    (d) none of the above.

6. Regarding the prelab,

    (a) you should do it before the lab.

    (b) you should do it during the lab.

    (c) you can skip it.

    (d) None of the above.

7. Regarding the lab results,

    (a) it should be handed the day of the experiment.

    (b) It should the signature from the instructor.

    (c) It can not be handed late.

    (d) All of the above.

8. I will not be allowed in the lab without my own safety goggles:

    (a) True

    (b) False

9. Where can you spiral-bind and double-side print the lab manual

    (a) Mcdonalds

    (b) Staples

    (c) Fedex

    (d) Staples and Fedex

10. This lab manual needs to be spiral-bind and double-side printed because

    (a) we need to save paper and trees

    (b) You will be tearing out some pages

    (c) All of the above

11. The two-signature rule says;

    (a) The pre-lab should be signed by your instructor in the lab.

    (b) The results of the experiment should be signed by your instructor in in the lab.

    (c) When handing the lab results it has to include two signatures from the instructor.

    (d) if you don't have two signatures you won't receive a grade as it means you did not do the work.

    (e) All of the above.

5 Hazard Symbols

Hazard symbols are recognizable symbols designed to warn about hazardous materials, locations, or objects. Identify the following signs and classify them according to the labels below. Identify any non-real hazard symbol. All Images CC-BY

- × Flammable, very flammable
- × Toxic
- × Very toxic
- × Irritant
- × Very irritant
- × Corrosive
- × Oxidizing
- × Explosive
- × Dangerous for the environment

A: _____

B: _____

C: _____
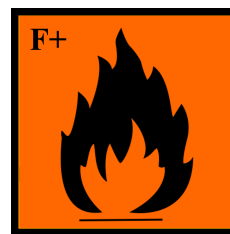
D: _____

E: _____

F: _____

G: _____

H: _____

I: _____

J: _____

NFPA 704 is a standard system for the identification of the hazards of materials for emergency response, This is a standard maintained by the U.S.-based National Fire Protection Association. It is commonly known as "Safety Square" or "Fire Diamond" and is used by emergency personnel to quickly and easily identify the risks posed by hazardous materials.

Gasoline
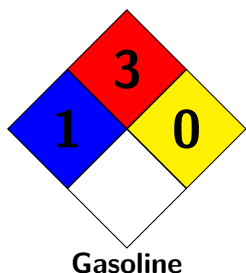
NFPA Code meaning					
Rating Number	Health Hazard	Flammability Hazard	Instability Hazard	Rating Symbol	Special Hazard
4	Can be lethal	Will vaporize and readily burn at room temperature	Might explode at normal conditions	OX	Oxidizing
3	Can cause serious or permanent injury	Can be ignited under almost all ambient temperatures	Might explode at high temperatures or shock	SA	Simple asphyxiants
2	Can cause temporary incapacitation or residual injury	Must be heated or high ambient temperature to burn	Violent chemical change at high temperature or pressure	W̶	Reacts violently with water
1	Can cause significant irritation	Can be preheated before ignition can occur	Normally stable hight temperatures make unstable		
0	No hazard	Will not burn	Stable		

HF

This chemical will burn. True or False?
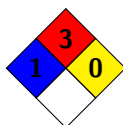
H$_2$CO$_3$

This chemical will burn your skin. True or False?

Acetic acid

This chemical will burn your skin. True or False?
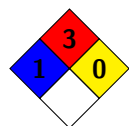
Acetone

This chemical will intoxicate you. True or False?
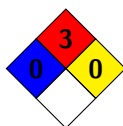
C$_2$H$_6$O

This chemical will burn. True or False?
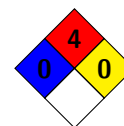
H$_{2(g)}$

This chemical will be reactive. True or False?

6   Some Lab Material

Identify the following laboratory equipment. Think about the basic use of each piece.

- × Dropper
- × Test tube
- × Measuring cylinder

- × Conical flask
- × Heating plate
- × buret

- × Watch glass
- × Beaker
- × Crucible
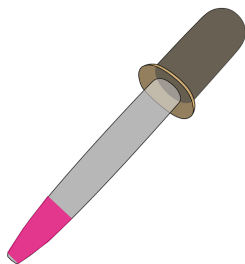
A
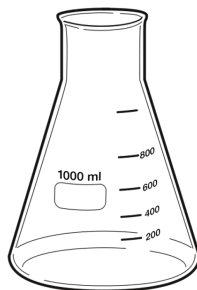
B
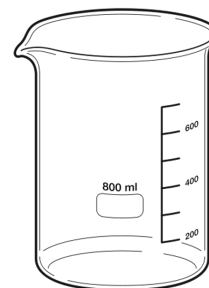
C

D

E
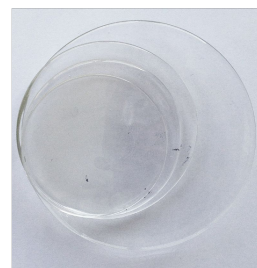
F
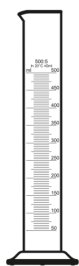
G

H

I

Search the lab and gather the following lab equipment. Show your instructor. Next, return each item to their original location:

- × Test tube rack
- × Crucible tongs
- × Clay triangle

- × Wire gauze
- × Buret
- × Suction Flask

- × Safety goggles

- × Beaker

EXPERIMENT

Identification of Unknown Compound: The scientific method

Goal

The goal of this experiment is to understand how the scientific method works. To do this you will make observations, look for patterns and come up with a hypothesis to *identify an unknown compound*. You will do this by comparing the reaction of the unknown compound with a set of known–reference–compounds, also called blanks. In this experiment, you will obtain some blank chemicals and an unknown. Your unknown is the same as one of the blanks. You will test each of these chemicals with a set of reagents. You have to observe the result of the test and record any change, such as *gas evolution*, the formation of a *precipitate*, a *change of color*, or maybe *no reaction*. Simultaneously, you will run similar tests for the unknown sample that is identical to one of the blanks. You will determine the unknown's identity by matching its characteristic reactions with one of the blanks.

Materials

- ☐ Six test tubes.

- ☐ The blanks: $NaCl(s)$, $NaI(s)$, $Na_2CO_3(s)$, $Na_2HPO_4(s)$ and $Na_2SO_4(s)$.

- ☐ Unknown compound.

- ☐ The reagents: $HNO_3(aq)$, $Ba(NO_3)_2(aq)$, $AgNO_3(aq)$ and thymol blue.

Background

Chemistry involves the in-depth study of matter and its transformations. The *macroscopic level* refers to the level at which we can actually see the changes happening with our own eyes, without a microscope. This level deals with the properties of large objects. On the other hand, the *microscopic level* refers to a deeper level that is inaccessible to the eye.

The scientific method: how science works

Scientists practice the *scientific method*–a set of steps–to make discoveries. The first step is to collect data by *making observations and measurements*. These measurements are usually made on a sample. The next step consists of *looking for patterns and trends* in the data. When a pattern is observed, scientists *develop a hypothesis*, that is a feasible explanation of the observations. After formulating a hypothesis, scientists think of further experiments to verify the hypothesis. If the results of repeated experiments support the hypothesis, scientists *formulate a theory* that explains the observations. One of the main goals of this experiment, adapted from the literature [1, 2], is to show you how chemists build models, turn them into a testable form, and then refine them in the light of additional evidence.

Reaction signals: gas evolution and precipitate reactions

Certain signs tell us that a chemical reaction has taken place. Oftentimes, the formation of bubbles is a sign of reaction. In general, when gases are formed from a solution, you will see a *large amount* of bubbles moving upward through the liquid. A color change might also be a sign of a chemical reaction.
Another sign of reaction is the formation of a precipitate, such as when lead(II) iodide (PbI_2), which is an insoluble yellow

solid, precipitates result of mixing lead(II) nitrate ($Pb(NO_3)_2$) and potassium iodide (KI) solutions. Precipitation, on the other hand, produces solids that can set at the bottom of the test tube, or when they are finely divided, a solid can stay in suspension, producing a milky appearance.

Chemical and physical properties

A *physical property* of a substance refers to a property that can be measured without altering the identity of the substance. Examples of physical properties are mass, temperature, melting point, hardness, color, state of matter (solid, liquid, or gas), and density. On the other hand, a *chemical property* is the ability of a chemical to transform into a different chemical. For example, certain acids in the presence of zinc change their nature producing hydrogen.

Compounds

A *compound* is a substance that consists of two or more different elements. A *binary compound* consists of only two elements, for example, water (H_2O) is a binary compound made of hydrogen (H) and oxygen (O). A *ternary compound*, on the other hand, is made of three different elements. For example, nitric acid (HNO_3) is a ternary compound made of hydrogen (H), nitrogen (N), and oxygen (O).

Ionic and covalent compounds

Compounds can be classified as *covalent* or *ionic* compounds. Ionic compounds are made of a mixture of a metal with a nonmetal. For example, sodium chloride, NaCl, is an ionic compound, knowing that sodium (Na) is a metal and chlorine (Cl) is a nonmetal. On the other hand, covalent compounds are made of combinations of nonmetals. As an example, water, H_2O, is a covalent compound, because both H and O are nonmetallic elements.

Compounds and chemical valence

The elements of the periodic table combine using their chemical valence. These are simple numbers that indicate the ratio of atoms in a molecule. For example, the valence of Mg is 2+ (we write this as Mg^{2+}), whereas the valence of Cl is 1− (we write this as Cl^-). When Mg^{2+} and Cl^- combine, they exchange their valences forming Mg_1Cl_2, which should be written as $MgCl_2$. Notice that the ones are always assumed and not displayed. The signs indicate which elements come first in the compound, and positive valences are arranged first whereas negative valences are arranged second. Sometimes, and because the subscripts are a ratio of atoms, one needs to simplify the valences and, for example, the result of combining Mg^{2+} and O^{2-} is Mg_2O_2, that is written as MgO. Some elements, typically transition metals, can have more than one valence. In that case, the valence has to be specified in the name using Roman numbers. Oftentimes, you will encounter polyatomic ions, such as $SO_4{}^{2-}$ or $NO_3{}^-$.

Example

Write down the formula resulting of combining the following pairs of ions: Ca^{2+} with F^-, and Ca^{2+} with $SO_4{}^{2-}$.

- -

Answer: The resulting formulas would be: CaF_2 and $CaSO_4$.

Ionic compound naming

Ionic compounds are named by a combination of two words, one for each ion in the formula. The first word refers to the name of the first ion, the one on the left, whereas the second word comes from the name of the second ion, the one on the right, with an *-ide* ending. For example, the name of NaCl is *sodium chloride*. Sodium is the name of the ion on the left, whereas chloride comes from the name chlorine with an *-ide* ending.

Covalent compound naming

Covalent compounds are named by a combination of two words making use of a prefix to indicate the number of atoms in the molecule. The first word refers to the name of the first element on the left in the compound formula, whereas the second word comes from the name of the second element, the element on the right, with an *-ide* ending. You need to use prefixes (mono, di, tri, tetra,...) to indicate the number of atoms of each element. For example, the name of CO_2 is *carbon dioxide*. Carbon is the name of the element C, whereas dioxide comes from the name oxygen with the *-ide* ending. The prefix *di-* in dioxide indicates that there are two oxygen atoms in the molecule.

Acids and bases

Acids in most cases contain a hydrogen atom, *H.* Examples of that are HCl and H_2SO_4, both of which are acids. The latter is also called an oxoacid because it contains oxygen. Oxoacid formulas are written starting with H and ending with O, and contain another element in between. Hydracids, on the other hand, do not contain oxygen. An example would be HCl which is named hydrochloric acid. The names of oxoacids and hydracids can be found in tables.

The most common bases contain *OH* in their formula. These bases are hydroxides, such as NaOH, $Ca(OH)_2$. Acids names are nonstandard and one needs to rely on naming tables. Bases on the other hand are named with the word *hydroxide.* For example, NaOH is named *sodium hydroxide.*

Oxo salts

Oxosalts are the results of the reaction of an oxoacid and a base. They normally contain three elements listed as a metal first, then a nonmetal such as N, S, or C, and ended with oxygen. $NaNO_3$ is an example of an oxosalt. HNO_3 is an oxoacid named as *nitric acid.* The ion resulting from the oxoacid, NO_3^- is called nitrate and hence $NaNO_3$ is named as *sodium nitrate.*

Example

Name the following compounds: $MgCl_2$, P_2O_3, HCl, KOH, H_2CO_3, and Na_2CO_3.

Answers: $MgCl_2$ is named magnesium chloride, whereas P_2O_3 is diphosphorus trioxide. HCl is hydrochloric acid, whereas KOH is potassium hydroxide. H_2CO_3 is an oxoacid named carbonic acid. Its corresponding ion would be CO_3^{2-}, named carbonate. Hence Na_2CO_3 is named sodium carbonate.

Table 1 List of oxoacids and hydracids

Oxoacids	Acid name	Anion	Anion name
$HMnO_4$	Permanganic acid	MnO_4^-	Permanganate
H_2CO_3	Carbonic acid	CO_3^{2-}	Carbonate
HNO_3	Nitric acid	NO_3^-	Nitrate
HNO_2	Nitrous acid	NO_2^-	Nitrite
H_3PO_4	Phosphoric acid	PO_4^{3-}	Phosphate
H_2SO_4	Sulfuric acid	SO_4^{2-}	Sulfate
H_2SO_3	Sulfurous acid	SO_3^{2-}	Sulfite
$H_2S_2O_2$	Thiosulfurous acid	$S_2O_2^{2-}$	Thiosulfite
$HClO_4$	Perchloric acid	ClO_4^-	Perchlorate
HIO_4	Periodic acid	IO_4^-	Periodate

Hydracids		Acid name
HCl		Hydrochloric acid
HBr		Hydrobromic acid
HF		Hydrofluoric acid
HI		Hydroiodic acid
HCN		Hydrocyanic acid

References

(1)   Ebbing, R. W.D. D., *Experiments in General Chemistry*; Houghton Mifflin Harcourt Publishing Company: 2004.

(2)   Beran, J. A., *Laboratory manual for principles of general chemistry*; John Wiley and Sons: 2010.

Procedure

Part A: Indicate your hypothesis

☐ *Step 1:* – Obtain the unknown compound, the blanks, and the reagents.

☐ *Step 2:* – Compare the unknown with the blanks and guess which blank looks like the unknown. That is your hypothesis. Write down the name of your guess on the results page.

☐ *Step 3:* – Wash a 100 mL graduated cylinder and 400 mL beaker. Place a pea-size portion of the unknown in the beaker, and add 150 mL of water using the cylinder to measure it. Swirl until all the solid has been totally or partially dissolved.

☐ *Step 4:* – Set aside the remaining portion of your unknown.

> **Good Lab Practice**
>
> ✌ Obtain directions from your lab instructor for discharging the solutions that you will use in this experiment.

Part B: Testing for gas

☐ *Step 5:* – In this first experiment you will use the solid version of the blanks, not the droppers. Obtain six test tubes. In each test tube, you will add a blank reagent in a solid state, that is, one blank per test tube. To each test tube, you will add a pea-size sample of a different blank in a solid state using its spatula. You will add the unknown in the sixth test tube. This way, place a pea-size solid sample of $NaCl$ in the first test tube. Add a pea-size sample of NaI in the second test tube, and so on with the rest of the blanks, Na_2CO_3, Na_2HPO_4, Na_2SO_4 and your solid unknown in the following test tubes. Mark your test tubes for recognition.

☐ *Step 6:* – Add five drops of HNO_3 ⚠ to each test tube and record your observations in the results table: *GE, for gas evolution, NGE for no gas evolution.*

☐ *Step 7:* – Discharge the solutions from the test tubes into the right waste container. Wash the test tubes, and rinse them with distilled water.

Part C: Testing for a barium precipitate

☐ *Step 8:* – For the following test will use only liquid samples in the dropper, not the powders. Use the solutions of $NaCl$, NaI, Na_2CO_3, Na_2HPO_4 and Na_2SO_4 found in the laboratory, and the solution of your unknown prepared in Step 3 for the remaining tests in this practice.

☐ *Step 9:* – Using six clean test tubes, add 20 drops of each of the solutions as well as the unknown to separate test tube. In short, five of the test tubes should have a different blank and the sixth test tube should have the unknown solution. Add 3 drops of a solution of ammonia (NH_3) in each test tube ⚠ and swirl until solved.

☐ *Step 10:* – Add five drops of the $Ba(NO_3)_2$ ⚠ solution to each of the test tubes. Shake gently to obtain homogeneity. Examine each test tube carefully and look at the results. Record your observations: *P for precipitate, NP for no precipitate.*

⚠ CAUTION!

⚠ Nitric acid (HNO_3) and ammonia (NH_3) ⚠ can cause chemical burns.

⚠ Solutions containing barium are toxic.

Part D: Testing for silver precipitate

☐ *Step 11:* – Using six clean test tubes, add 20 drops of each of the solutions including the unknown to separate test tubes (e.g. NaCl, NaI, Na_2CO_3, Na_2HPO_4, Na_2SO_4, and unknown). In short, five of the test tubes should have a different blank and the sixth test tube should have the unknown solution.

☐ *Step 12:* – Add five drops of the nitric acid $AgNO_3$ solution to each test tube. Record your observations: *P for precipitate, NP for no precipitate.* Include the color of the precipitate.

Part E: Testing with thymol blue

☐ *Step 13:* – Using six clean test tubes, add 20 drops of each of the solutions including the unknown to separate test tubes (e.g. NaCl, NaI, Na_2CO_3, Na_2HPO_4, Na_2SO_4, and unknown). In short, five of the test tubes should have a different blank and the sixth test tube should have the unknown solution.

☐ *Step 14:* – Add five drops of the thymol blue solution to each test tube. Shake each test tube gently before recording your observations: *write down Blue, Yellow green, or Red.*

Part F: Identifying the unknown compound

☐ *Step 15:* – The unknown is one of these compounds: NaCl, NaI, Na_2CO_3, Na_2HPO_4, Na_2SO_4. You should be able to identify your unknown compound by matching its reactions with those of one of the known samples.

☐ *Step 16:* – When done with the experiment, clean up all test tubes using a test tube brush and soapy water.

Pre-lab Questions

Identification of Unknown Compound: The scientific method

1. Classify the following compounds as *ionic, covalent, acids, bases* or *oxo salts*:

   NH_3 _____ SO_3 _____

   HNO_3 _____ NaF _____

2. Give the names or formulas for the following compounds:

   NaCl _____ HNO_3 _____

   Sodium iodide _____ Sodium carbonate _____

3. Name or give the formula of the following compounds:

   SO_2 _____ NO_2 _____

   Lithium fluoride _____ Sodium oxide _____

4. What does a pea size of solid mean?

5. Where in the lab you will be disposing of the disposals from this experiment?

6. The following table resulted from an experiment, very similar to the one you are going to carry. The goal was to identify an unknown compound between KCl and KI. Look at the reactivity patterns and identify the unknown.

		Reagents			
		HNO_3	$Ba(NO_3)_2$	$AgNO_3$	Thymol Blue
Blanks	KCl	NP	NP	P (white)	Green
	KI	NP	NP	NP	Green
	Unknown	NP	NP	NP	Green

My unknown is = _____

Results
EXPERIMENT

Identification of Unknown Compound: The scientific method

Unknown Label # = _____ Your guess = _____

		Reagents			
		HNO_3	$Ba(NO_3)_2$	$AgNO_3$	Thymol Blue
Blanks	NaCl				
	NaI				
	Na_2CO_3				
	Na_2HPO_4				
	Na_2SO_4				
	Unknown				

(write GE for gas evolution, NGE for no gas evolution, P for precipitate, NP for no precipitate and Blue, Yellow or Green for the color for the Thymol blue experiment)

My unknown is = _____

Post-lab Questions

Identification of Unknown Compound: The scientific method

1. Classify the following compounds as *ionic, covalent, acids, bases* or *oxo salts*:

    $Ba(OH)_2$ _____ CO_2 _____

    HCl _____ H_2SO_4 _____

    LiCl _____   AuCl _____

    Na_2SO_4 _____   NaOH _____

2. Give the names or formulas for the following compounds:

    HNO_3 _____   Sodium sulfate _____

    Barium nitrate _____ Na_2CO_3 _____

    $AgNO_3$ _____   Nitric acid _____

3. Name or give the formula of the following compounds:

    $CuCl_2$ _____   Hydrogen chloride _____

    Nickel (II) chloride _____   BaO _____

    CoO _____   MgO _____

    Sodium iodide _____   Potassium iodide _____

    NH_3 _____   LiH _____

8 Measurements of Mass and Volume

EXPERIMENT

Measurements of Mass and Volume

Goal

The goal of this experiment is to determine the *precision* of your measurements and to learn how to express correctly a *measured* result. You will learn also how to use a pipet and how to determine the precision of your measured volume. You will do this by repeatedly adding water from a filled pipet into a flask, and then measuring the mass of the liquid added to the flask with a scale. Knowing the density of water, you will be able to calculate the volume delivered into the flask. Next, you will repeat the process using a graduated cylinder and compare the precision of the two methods. You will also carry simple statistics on the measured results.

Materials

- ☐ 250 mL beaker
- ☐ 10 mL volumetric pipet, 10ml graduated pipet and 10 mL cylinder
- ☐ Thermometer

- ☐ 50 mL Erlenmeyer flask with a rubber stopper
- ☐ Analytical balance
- ☐ Tab water

Background

Most chemistry experiments require the measurement of a property (mass, volume, temperature, color...), and the validity of those experiments will depend on the reliability of each measurement. The reliability of a measurement is usually considered in terms of its *accuracy* and its *precision*. The *accuracy* of a measurement is the degree of closeness of measurements of a quantity to that quantity's true, exact, value. On the other hand, the *precision* of a measurement refers to the reproducibility of the experiment, that is the degree to which repeated measurements show the same results. This is a very simple and insightful experiment, implemented in many different forms in the chemistry lab literature over the years [1–4].

Significant Figures

The digits of a quantity that has been experimentally measured are not exact, and every measurement has a certain degree of uncertainty, which depends on the device used for the measurement. We should take the uncertainty in our measurements into account to know the error associated with them. One way to do this is to report the result of a calculation with the correct number of *significant figures* (SF). The digits different than zero are always significant. Differently, the zeros can or can not be significant. To identify the correct number of significant zeros of a quantity, one needs to follow these four rules:

(a) *Rule one:* zeros at the beginning of a decimal number (these are called leading zeros) are not significant: 0.123 has 3 SF.

(b) *Rule two:* terminal zeros at the right of the decimal point (these are called trailing zeros) are significant: 0.120 has 3 SF

(c) *Rule three:* terminal zeros in a whole number (a number without an explicit decimal point) are not significant: 1230 has 3 SF.

(d) *Rule four:* for a number in scientific notation, all numbers before the power of ten are significant: 1.50×10^{-5} has 3 SF.

The number of significant figures in a measured property is determined by the tool employed in the measurement. When the data is used in subsequent calculations the uncertainty must be carried on with the results. The following two rules hold when determining the correct number of significant figures to report for the answer to a calculation:

(a) *Rule one:* when *multiplying or dividing* measured quantities, give as many significant figures in the answer as there are in the measurement with the *least number of significant figures.*

(b) *Rule two:* when *adding or subtracting* measured quantities, give the same number of decimal places in the answer as there are in the measurement with the *least number of decimal places.*

Example

Indicate the number of significant figures (SF) for the following values; (a) and (b), and perform the following calculations; (c) and (d), rounding the answers to the correct number of significant figures:

(a) 0.1250

(b) 56,700

(c) $\dfrac{1.235 \times 3.2}{2.34}$

(d) $1.235 + 3.2$

- -

Answers: (a) 0.1250 has 4 SF.   (b) 56,700 has 3 SF.   (c) The number with the fewest significant figures is 3.2; 2 SF. Hence, the result of the calculation, 1.68889, should be rounded to two significant figures; 1.7   (d) The lowest place value in common for the 2 amounts is the tenths, hence the result of the addition, 4.435, should be rounded to one decimal place; 4.4

Average value and Standard deviation

If we measure a property repeatedly, some measurements will be closer to the true, exact, value than others. This is because there can be variations between the amount of the property being measured and in the actual measurement itself. This variability contributes to the dispersion in the measured values. The general approach is to perform a number, n, of replicated measurements under similar conditions. Obtaining several measurements allows us to calculate the sample *mean value*, $\bar{x}$, and the *standard deviation*, σ.

The sample mean $\bar{x}$ is the average value for a finite set of replicate measurements on a sample and is calculated using the formula:

$$\bar{x} = \frac{1}{n} \sum_i^n x_i$$

where $\sum_i^n x_i$ represents the sum of all measurements, x. The sample standard deviation σ provides estimates of the population values and it is calculated using the formula:

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

Reporting measured results

The final value for a measurement should be written as:

$$x = \bar{x} \pm \sigma$$

notice that the standard deviation should have the same number of decimals as the average value. Read the following example to see how the calculation is done with real numbers.

Example

We measure one sample mass several times and obtain the values: 108.6 g, 104.2 g, 96.1 g, 99.6 g, and 102.2 g.

(a) *Compute the average, $\bar{x}$.*

(b) *Compute the standard deviation of the measurement, σ.*

(c) Report the measured mass in the form $\bar{x} \pm \sigma$.

- -

Answer: (a) The average mass is given by:

$$\bar{m} \;=\; \tfrac{1}{5}(108.6\ g \;+\; 104.2\ g \;+\; 96.1\ g \;+\; 99.6\ g \;+\; 102.2\ g) \;=\; 102.1\ g.$$

(b) The standard deviation is given by:

$$\sigma = \sqrt{\tfrac{1}{4}\Big((108.6\,g - 102.1\,g)^2 + (104.2\,g - 102.1\,g)^2 + (96.1\,g - 102.1\,g)^2 + (99.6\,g - 102.1\,g)^2 + (102.2\,g - 102.1\,g)^2\Big)} = 4.7\,g.$$

(c) The measured mass is given by: $102.1 \pm 4.7\ g$

Use of analytical balance and volumetric material

An *analytical balance* is a class of balance designed to measure small masses. The measuring pan of an analytical balance is inside a transparent container with doors to avoid dust and air currents. On the other hand, the volumetric measurement of liquids is a very common task in the laboratory, and volumetric instruments, such as volumetric flasks, bulb pipettes, graduated pipettes, graduated cylinders and burettes are standard laboratory equipment. On the contrary, graduated beakers, beakers, Erlenmeyer flasks, dropping funnels, and the like containers *are not volumetric instruments*. They are not precisely calibrated, and the volume marks serve only as approximate guides. Transfer pipets are sensitive volumetric materials calibrated to deliver a specific volume of a liquid.

Correct meniscus setting is a must to accurately measure a volume. To read the correct volume, the volumetric instrument should be held upright and the observer's eye must be at the same height as the meniscus. In this position, the ring mark on the measuring tool will flatten into a straight, horizontal line. For a better reading, the meniscus will turn up darker and more easily readable in front of a light background if a piece of dark paper is held behind the instrument immediately beneath the ring mark or graduation mark.

Density

The density of an object (d) is its mass (m) per unit volume (V). You can express this as:

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

Density is an important physical property of a material. The value of density changes with the temperature. Water, for example, has a density of $1.000\,g/mL$ at $4.0°C$ and a density of $0.998\,g/mL$ at $20.0°C$.

References

(1)   Ebbing, R. W.D. D., *Experiments in General Chemistry*; Houghton Mifflin Harcourt Publishing Company: 2004.

(2)   Brescia, F., *Fundamentals of Chemistry Laboratory Studies*; Elsevier: 1966.

(3)   Sawyer, C. P.E.H. D., *Quantitative Analysis*; Wiley: 1948.

(4)   NIST., $\mathtt{https://www.nist.gov/sites/default/files/documents/calibrations/74\text{-}461.pdf}$, Accessed: 2017-04-22.

Procedure

Getting started

☐ *Step 1:* – Obtain 100 mL of water in a beaker, a 10 mL pipet, a thermometer, and a 50 mL Erlenmeyer flask with a rubber stopper.

☐ *Step 2:* – Record the temperature of the water you have set aside with the thermometer.

☐ *Step 3:* – Find out the water density for that temperature by visiting the following website: ❋ `https://www.internetchemistry.com/chemical-data/water-density-table.php`. Copy the result with four significant figures.

☐ *Step 4:* – Locate an analytical scale in the lab. Check the bubble of the leveling eye to make the scale pan flat. If it's not, adjust the corners of the scale until the bubble enters the inner circle.

Good Lab Practice

✌ Do not handle objects to be weighed with bare hands, as moisture, grease, and dirt on your fingers will affect the weight of the objects.

Part A: Volume using a pipet

☐ *Step 1:* – Practice pipetting liquid several times, until you are comfortable with the technique.

☐ *Step 2:* – Bring the balance to zero and measure the mass of the dry Erlenmeyer with the stopper on. Write down the mass in the Results table always including all decimals given by the scale.

☐ *Step 3:* – Remove the stopper and pipet 10.00 mL of the room-temperature water into the flask.

☐ *Step 4:* – Bring the balance to zero and measure and record the combined mass of the water and the stoppered flask always including all decimals given by the scale.

☐ *Step 5:* – Remove the flask from the balance and remove the stopper. *Do not empty the flask.* Add another 10.00 mL sample into the flask, leaving the first water sample in the flask. The volume of water in the flask should now be approximately 20 mL. Put back the stopper and record the mass of the water and the stoppered flask copying all decimals provided by the scale.

☐ *Step 6:* – Keep pipetting 10.00 mL of water and recording the mass until four samples of water have been delivered to the flask and the final volume is 40.00 mL.

☐ *Step 7:* – The mass of water that was delivered each time from your pipet, is the difference between masses after each addition. Calculate the mass of water added by subtraction. These masses should be quite similar.

☐ *Step 8:* – Using the water density from your records, calculate the volume of each water sample.

Part B: Volume using a cylinder

☐ *Step 1:* – Practice reading the meniscus of a 10mL cylinder filled with water until you are comfortable with the technique.

☐ *Step 2:* – With a thermometer, record *again* the temperature of the water you have set aside, and write it down in the Results sheet.

☐ *Step 3:* – Find out the water density for that temperature by visiting the following website: ❋ `https://www.internetchemistry.com/chemical-data/water-density-table.php`

☐ *Step 4:* – Bring the balance to zero and measure and record the mass of *the same, empty, dry* Erlenmeyer with the stopper on.

☐ *Step 5:* – Using tissue paper and without touching the flask, remove the stopper and add to the flask *exactly* 10.00 mL of the room-temperature water using the cylinder.

☐ *Step 6:* – Measure and record the combined mass of the water and the stoppered flask.

☐ *Step 7:* – Remove the flask from the balance and remove the stopper. Add then another 10.00 mL of water into the flask, leaving the first water sample in the flask. The volume of water in the flask should now be approximately 20 mL. Replace the stopper and record the mass of the water and the stoppered flask.

☐ *Step 8:* – Keep adding 10.00 mL of water and recording the mass until four samples of water have been delivered to the flask and the final volume is 40.00 mL.

☐ *Step 9:* – The mass of water that was delivered each time from your cylinder, is the difference between masses after each addition. Calculate the mass of water added by subtraction. These masses should be quite similar.

☐ *Step 10:* – Using the water density from your records, calculate the volume of each water sample.

Part C: Volume using a graduated pipet

☐ *Step 1:* – With a thermometer, record *again* the temperature of the water you have set aside, and write it down in the Results sheet. Find out the water density for that temperature by visiting the following website:
✦ `https://www.internetchemistry.com/chemical-data/water-density-table.php`

☐ *Step 2:* – Bring the balance to zero and measure and record the mass of *the same, empty, dry* Erlenmeyer with the stopper on.

☐ *Step 3:* – Using tissue paper and without touching the flask, remove the stopper and add to the flask *exactly* 10.00 mL of the room-temperature water using a graduated pipet. To add 10mL you need to fill the pipet to the zeroth mark and let the liquid go until the liquid level reaches the 10mL mark.

☐ *Step 4:* – Measure and record the combined mass of the water and the stoppered flask.

☐ *Step 5:* – Remove the flask from the balance and remove the stopper. Add then another 10.00 mL of water into the flask, leaving the first water sample in the flask. The volume of water in the flask should now be approximately 20 mL. Replace the stopper and record the mass of the water and the stoppered flask.

☐ *Step 6:* – Keep adding 10.00 mL of water and recording the mass until four samples of water have been delivered to the flask and the final volume is 40.00 mL.

☐ *Step 7:* – The mass of water that was delivered each time from your pipet, is the difference between masses after each addition. Calculate the mass of water added by subtraction. These masses should be quite similar.

☐ *Step 8:* – Using the water density from your records, calculate the volume of each water sample.

Calculations

⓪ Obtain the density of water at the measured temperatures using the link below:

✦ `https://www.internetchemistry.com/chemical-data/water-density-table.php`

① The mass added of water is calculated using:

(mass of water after the addition) − (mass of water before the addition)

② The volume of water in each addition is:
$$v_i = \boxed{1} / \; d$$

where d is the density of water at the lab temperature and m_i is the mass of water added to the beaker.

③ The average volume of the four measurements can be computed using the formula:

$$\bar{v} \quad = \quad \tfrac{1}{4}(v_1 \ + \ v_2 \ + \ v_3 \ + \ v_4)$$

(4) The standard deviation in the volume measurements can be computed using the formula:

$$\sigma = \sqrt{\frac{1}{3}\left[(v_1 - \bar{v})^2 + (v_2 - \bar{v})^2 + (v_3 - \bar{v})^2 + (v_4 - \bar{v})^2\right]}$$

where $\bar{v}$ is the average volume calculated in the previous calculation step. You can also use the link below to calculate the standard deviation. Make sure you select the "sample" option:

🌐 `https://www.calculator.net/standard-deviation-calculator.html`

Pre-lab Questions

Measurements of Mass and Volume

1. Throughout this experiment you will use tap water. How different would the results be if you had used distilled water instead?

2. Indicate the measurement of the following meniscus:
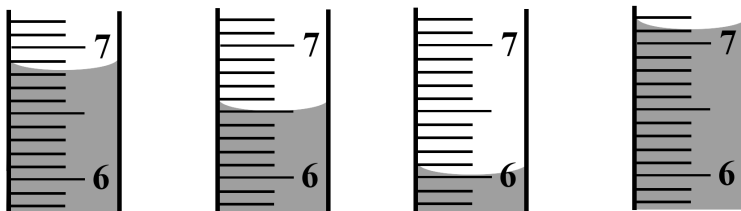
3. Express the answer to the following calculations to the a correct number of significant figures:

   (a)
   $$\frac{60.875 \times 2.700}{60.4 + 107.1} =$$

   (b)
   $$\frac{(604.01 + 0.73) \times 321.81 \times 0.00480}{3.630 \times 10^{-3}} =$$

4. We measure the mass of an object four times and the results obtained in grams are: 2.092, 2.100, 2.053, 1.998g.

   (a) Calculate the average mass $\bar{m}$ with the right number or significant figures.

   (b) Calculate the standard deviation σ of the mass measurements.

   (c) Express the measured value of the mass in the form: $\bar{m} \pm \sigma$, and indicate how many significant figures are justified in the measured value.

5. Calculate the water density at 35.0°C by visiting the following website: ☀ https://www.internetchemistry.com/chemical-data/water-density-table.php

6. Determine what mass of water is delivered from a 10.00 mL pipet at $39.0°C$ if the density of water at that temperature is 0.9918 g/mL.

Results
EXPERIMENT

Measurements of Mass and Volume

Temperature (°C)=——————————————————Density of water (g/mL) ⓪ =————————————————————

Part A: Using the Pipet				
Addition Number	1	2	3	4
Mass before addition (g)				
Mass after addition (g)				
① Mass of added water (g)				
② Volume of water delivered (mL)				
③ Average Volume delivered, $\bar{v}$ (mL)				
④ Standard Deviation, σ (mL)				
$\bar{v} \pm \sigma$ (mL)				

(The boxes connected with an arrow should contain the same information)

Temperature (°C)=_____Density of water (g/mL) $\boxed{0}$ =_____

Part B: Using the Cylinder					
	Addition Number	1	2	3	4
	Mass before addition (g)				
	Mass after addition (g)				
①	Mass of added water (g)				
②	Volume of water delivered (mL)				
③	Average Volume delivered, $\bar{v}$ (mL)				
④	Standard Deviation, σ (mL)				
	$\bar{v} \pm \sigma$ (mL)				

The boxes connected with an arrow should contain the same information)

Temperature (°C)=_____Density of water (g/mL) $\boxed{0}$ =_____

Part C: Using the graduated pipet					
	Addition Number	1	2	3	4
	Mass before addition (g)				
	Mass after addition (g)				
①	Mass of added water (g)				
②	Volume of water delivered (mL)				
③	Average Volume delivered, $\bar{v}$ (mL)				
④	Standard Deviation, σ (mL)				
	$\bar{v} \pm \sigma$ (mL)				

STUDENT INFO

Name: Date:

Post-lab Questions

Measurements of Mass and Volume

1. The results of measuring the mass of an *stopped Erlenmeyer flask* mass are 50.123g, 50.013g, 49.933g, and 51.002g,

   (a) Calculate the standard deviation in your measured mass for the empty stopped flask.

   (b) How does the result compare with the claimed precision of your balance? The balance precision is ± 0.0001 g.

   (c) Express the measured value of the mass in the form: $\bar{m} \pm \sigma$.

2. For the measurement of the *volume of water in a pipet:* compare the precision of the pipet with your result. The pipet precision is ± 0.01 mL.

3. Compare the accuracy of the volume measurements when using a pipet with the one when using a cylinder.

4. Compare the precision of the volume measurements when using a pipet with the one when using a cylinder.

5. Using the data from:
   🌐 https://www.internetchemistry.com/chemical-data/water-density-table.php
   plot the values of density vs. temperature for water: (use a range between 0.0°C and 90.0°C)
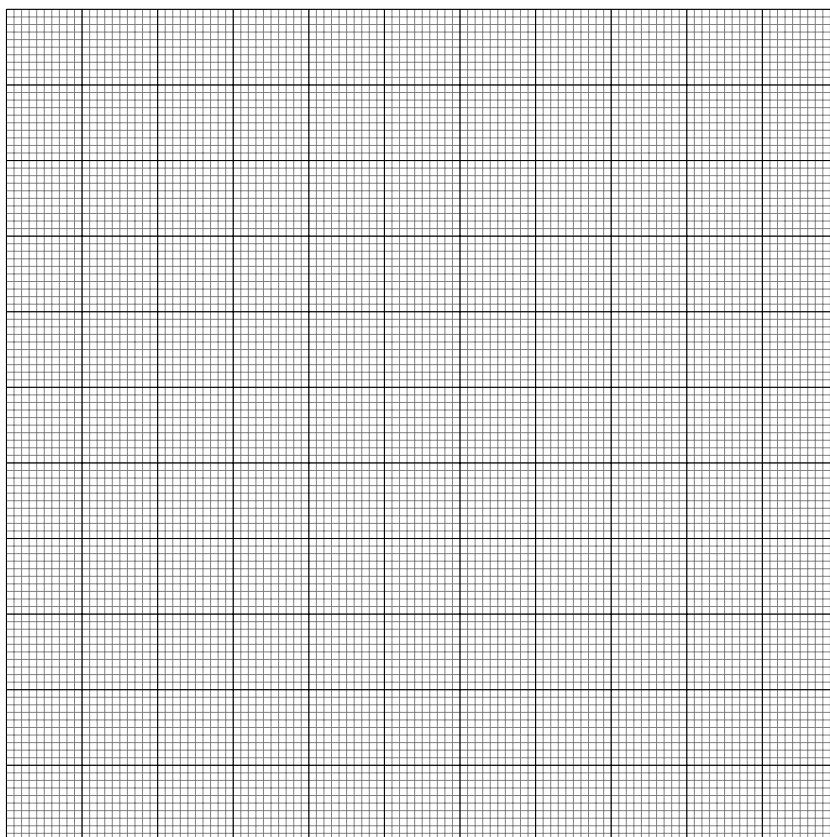
6. The darts in these targets represent measurements that are precise and accurate (PaA), precise but inaccurate (PbI), imprecise but accurate on average (IbA), and both imprecise and inaccurate (IaI). Correlate the targets with the most suitable description.
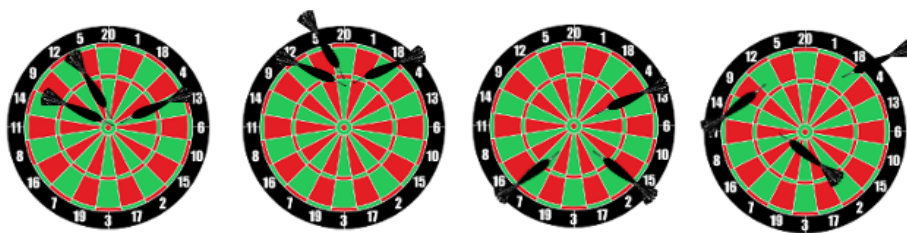
EXPERIMENT

Isotopes and Mass Spectroscopy

Goal

The goal of this experiment is to determine the *isotopic composition* and *atomic masses* of several elements. You will achieve this goal by studying mass spectra. By measuring the height of the mass spectrum peaks–as the height of a signal is proportional to the fractional abundance–you will be able to calculate the fractional abundances of the different isotopes.

Materials

This is a modeling-based experiment, and you will not need a real mass spectrometer to experiment. You will only need a ruler and the mass spectra provided here and extracted from ☙ *www.webelements.com* and the ☙ *National Institute of Standards and technology*, a US measurement standards laboratory.

Background

A mass spectrometer is an instrument used to determine atomic and molecular masses. This instrument can also be used to determine the masses and isotopic composition of an element. In this theory-based experiment, adapted from the literature [1], you will first analyze a set of mass spectra. Then, from their peaks, you will calculate the isotopic composition of a series of elements.

The nuclear model

According to the current nuclear model of the *atom*, atoms contain a small positively charged nucleus, which is responsible for almost all atomic mass, and a negatively charged electronic cloud. When the positive charge of the nucleus exactly cancels the negative charge of the surrounding electrons, we say that atoms are electrically neutral. The positively charged particles inside the nucleus are called *protons* (denoted p), whereas *electrons* (denoted e^-) have a single negative charge. The number of protons of an element is called the *atomic number*, Z, of that element. For neutral atoms, the number of protons equals the number of electrons. As an example, the nucleus of a hydrogen atom (H) has one proton, so its atomic number is $Z=1$, whereas the nucleus of a helium atom (He) has two protons, so its atomic number is $Z=2$. The atomic nucleus also contains electrically neutral particles called *neutrons* (denoted n). Neutrons have approximately the same mass as protons, and so they add substantially to the mass of the nucleus. Therefore, differences in the number of neutrons in a nucleus give rise to different atomic masses, even though those atoms are of the same element. Apart from their charge, neutrons, and protons are jointly known as *nucleons*.

Isotopes and atomic mass

The total number of protons and neutrons in a nucleus is called the *mass number, A*, of the atom. All the atoms of a particular element have the same atomic number. However, different atoms of the same element can have different mass numbers. *Isotopes* are atoms of the same element that have the same atomic number but a different mass number. Different isotopes have different masses and different nuclear properties. Isotopes from the same element are differentiated by writing their mass number after the name of the element, as in neon-20, neon-21, and neon-22. Its symbol such as $^A_Z X$ is obtained by writing the mass number A as a superscript to the left of the chemical symbol of the element and the atomic number Z included as a subscript on the lower left, as in $^{20}_{10}Ne$. Some isotopes of an atom are more common than others and this is

quantified using the isotope *percent abundance, F*. For example, hydrogen has three isotopes. The most common is $^1_1$H called protium with a 99.985% abundance. The second isotope, deuterium $^2_1$H, is less common, with 0.015% abundance. The third one called tritium is a very rare isotope. The abundance can also be expressed as a *fractional abundance f* and for example the fractional abundance of $^1_1$H is 0.99985.

The *atomic mass* of an element, expressed in *amu* (atomic mass units), is the weighted average of the masses of the individual isotopes of the element. For an element with n isotopes with different masses (A_1, A_2, ..., A_n) and different fractional abundances for each isotope (f_1, f_2, .., f_n), the atomic mass is given by

$$\text{Atomic mass} = \sum_{i=1}^{n} A_i \cdot f_i$$

Example

Naturally occurring copper (Cu) consists of 69.17% ^{63}Cu and 30.83% ^{65}Cu. The mass of ^{63}Cu is 62.939598 amu, and the mass of ^{65}Cu is 64.927793 amu. What is the atomic mass of copper?

- -

Answer: The weighted average is the sum of the mass of each isotope times its fractional abundance:

$$62.939598 \, amu \times \frac{69.17}{100.} + 64.927793 \, amu \times \frac{30.83}{100.} = 63.55 \, amu$$

Mass spectrometry

Mass spectrometry is a very powerful analytical technique that provides both quantitative and qualitative information about the chemical being analyzed. In a mass spectrometer, the sample is first vaporized to generate atoms and then introduced into the sample inlet chamber which leads to an ionization chamber. In this chamber, electrons collide with the vaporized species producing ions and new electrons. The resulting ions are accelerated by an electric field into a beam that is deflected toward a detector by a magnetic field. The heavier the particle, the lower the bean deflection. Hence, the degree of deflection is related to the relative masses of the deflected particles.

Mass spectrum

The positions of the peaks in the mass spectrum give the mass-to-charge ratios of the ions. If the sample consists of atoms of one element, the mass spectrum gives the isotopic distribution of the sample. Mass spectra of molecules are more complex as bond breaking can occur. Thus the mass spectrum of a molecule contains signals that are due to fragments of molecules in the mass spectrum, as well as those that are due to the unfragmented molecule. The mass of this molecular ion, the parent ion, is called the parent mass. The parent ion has essentially the same molar mass as the compound itself.

Example

Calculate the % abundance of the naturally occurring isotopes of Ne from the lines of the spectrum below. Assume the length of the smallest pick is 0.05cm.
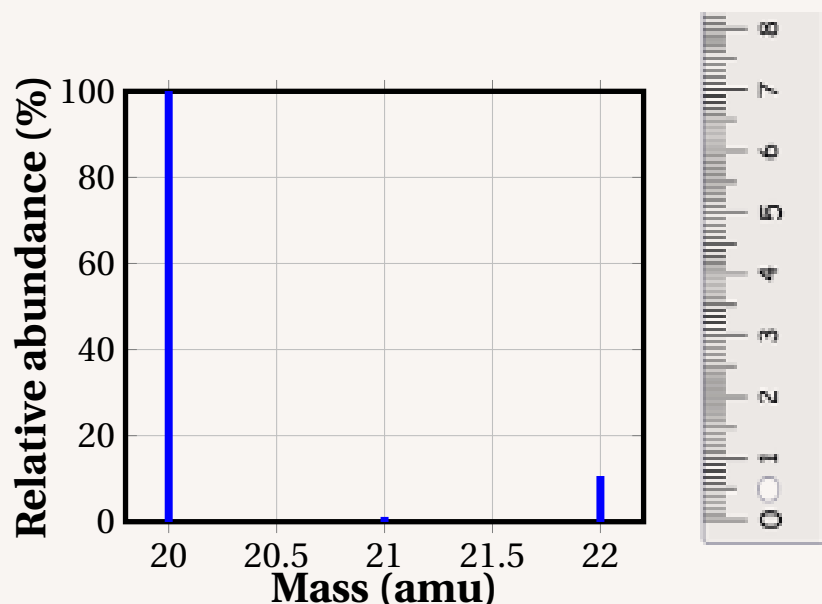
Answer: the mass spectrum of He consists of an intense signal at mass number 20, a much smaller signal at a mass of 22, and a very weak signal at 21 amu. The horizontal coordinates of each peak represent the mass number of each isotope (A_i), as listed in the table below. The heights of these peaks are proportional to the number of counts at each mass number and, in return, reflect the fractional abundances of the isotopes. By using a ruler we can measure the length of the peaks, as we list them in the table below. By adding the length of each peak we obtain I_{total}, also listed in the table. We obtain the fractional abundance of each isotope f_i by dividing the intensity of each peak by the total intensity, also listed in the table below. The fractional abundance is related to the isotope abundance. For example, the fractional abundance of $^{22}_{9}$Ne is 0.903, hence, the abundance of this isotope is 90.3%.

Isotope ($^{A_i}_{Z}$X)	Mass Number (A_i)	Intensity (I_i)	Fractional Abundance (f_i)	Isotope contribution ($f_i \cdot A_i$)
^{20}Ne	20	7.00 cm	$\frac{7.00}{7.75} = 0.903$	$0.903 \cdot 20 = 18$ amu
^{21}Ne	21	0.05 cm	$\frac{0.05}{7.75} = 0.006$	$0.006 \cdot 21 = 0.1$ amu
^{22}Ne	22	0.70 cm	$\frac{0.70}{7.75} = 0.090$	$0.090 \cdot 22 = 2.0$ amu
	$I_{total} = 7.00 + 0.70 + 0.05 = 7.75$ cm		$AW_{experimental} =$	$18 + 0.1 + 2.0 = 20.1$ amu
			$AW_{Theoretical} =$	20.2 amu
			% Error AW =	0.49%

Finally, we obtain the isotope contribution to the atomic mass of the element by multiplying the fractional abundance by the mass number of each isotope. By adding all isotope contributions we obtain the average atomic mass, which can be compared from the value found on the periodic table. The error between the calculated and experimental values is less than 5%.

References

(1)   Ebbing, R. W.D. D., *Experiments in General Chemistry*; Houghton Mifflin Harcourt Publishing Company: 2004.

Calculations

The mass spectrum of a series of atoms is provided next page. Select each of the spectra, while following the next steps:

(0) Pick up one of the mass spectra. Write down the name of the element.

(1) Write down the name of the isotope. Z is the atomic mass from the periodic table. A is the mass number of the pick. X is the name of the element.

(2) Write down the mass number of each pick (horizontal reading of the mass spectra).

(3) With a ruler measure the length of each pick. Consider the precision of your ruler and use the last decimal possible (estimated value). Use either cm or in.

(4) Add the intensity of each pick.

(5) Calculate the fractional abundance by doing: (3)/(4)

(6) Calculate the isotope abundance: (5) × (2)

(7) Calculate the average atomic mass by adding all isotope contributions without rounding the AW.

(8) Obtain the theoretical atomic mass of the element from the periodic table

(9) Calculate the % Error AW as indicate below. The value should be positive. If the error is higher than 10% you need to review the accuracy of your measurements and redo the numbers:

$$\% \text{ Error AW} = \left| (7) - (8) \right| / (8) \times 100\%$$
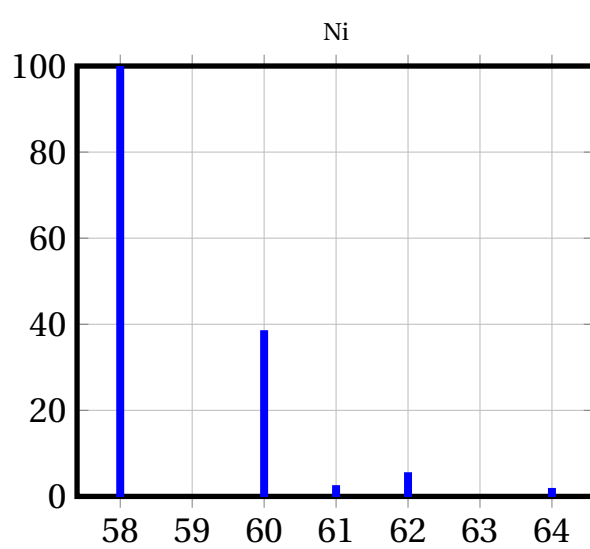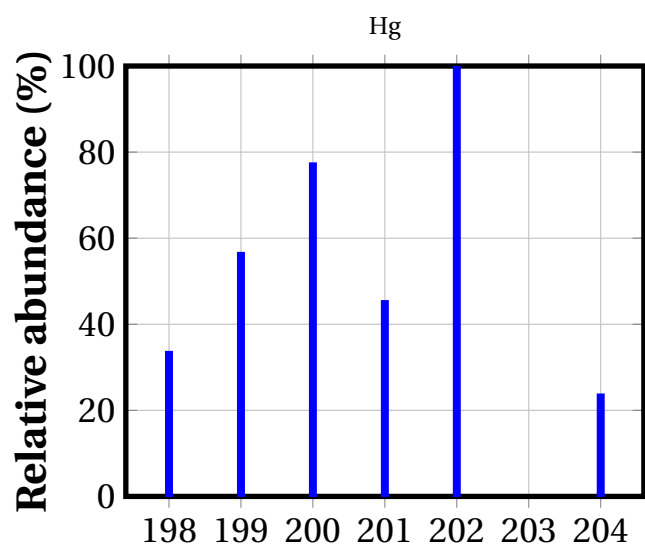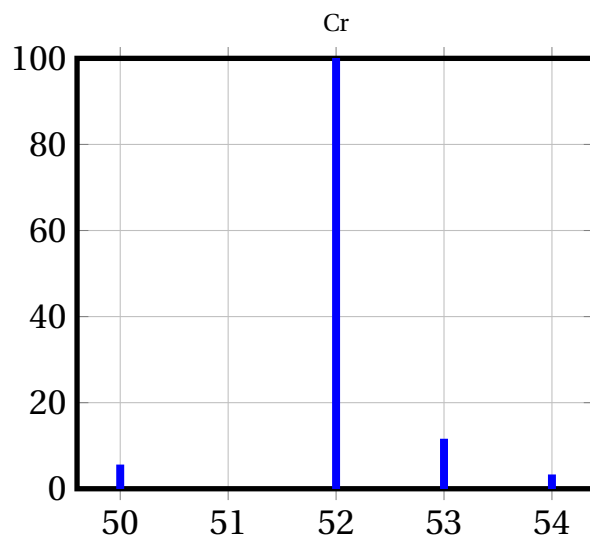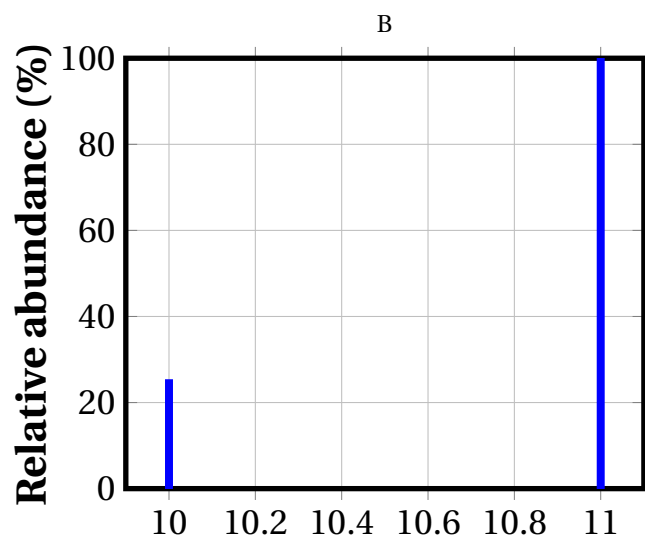
STUDENT INFO

Name: Date:

Pre-lab Questions

Isotopes and Mass Spectroscopy

1. Consider the mass spectrum below of an element X: how many isotopes are present and what is the fractional abundance of the isotopes? Given that the average atomic mass of the element from the periodic table is 17.898 amu, use the table below to indicate the experimental atomic weight and the percent error.

Isotope ($^{A_i}_{Z}X$)	Mass Number (A_i)	Intensity (I_i)	Fractional Abundance (f_i)	Isotope contribution ($f_i \cdot A_i$)
	$I_{total} =$		$AW_{experimental}$	
			$AW_{Theoretical} =$	
			% Error AW =	

Results
EXPERIMENT

Isotopes and Mass Spectroscopy

⓪ Element=				
Isotope ($^{A_i}_{Z}X$) ①	Mass Number (A_i) ②	Intensity (I_i) ③	Fractional Abundance (f_i) ⑤	Isotope contribution ($f_i \cdot A_i$) ⑥
	$I_{total} =$ ④		$AW_{experimental}$ ⑦	
			$AW_{Theoretical} =$ ⑧	
			% Error AW = ⑨	

⓪ Element=				
Isotope $({}^{A_i}_{Z}X)$ ①	Mass Number (A_i) ②	Intensity (I_i) ③	Fractional Abundance (f_i) ⑤	Isotope contribution $(f_i \cdot A_i)$ ⑥
	$I_{total} =$ ④		$AW_{experimental}$ ⑦	
			$AW_{Theoretical} =$ ⑧	
			% Error AW = ⑨	

(0) Element=				
Isotope ($_Z^{A_i}$X) (1)	Mass Number (A_i) (2)	Intensity (I_i) (3)	Fractional Abundance (f_i) (5)	Isotope contribution ($f_i \cdot A_i$) (6)
	I_{total} = (4)		$AW_{experimental}$ (7)	
			$AW_{Theoretical}$ = (8)	
			% Error AW = (9)	

Isotope $(^{A_i}_{Z}X)$	Mass Number (A_i)	Intensity (I_i)	Fractional Abundance (f_i)	Isotope contribution $(f_i \cdot A_i)$
			(0) Element=	
(1)	(2)	(3)	(5)	(6)
	$I_{total} =$ (4)		$AW_{experimental}$ (7)	
			$AW_{Theoretical} =$ (8)	
			% Error AW = (9)	

Post-lab Questions

Isotopes and Mass Spectroscopy

1. The atomic mass of Ga is 69.72 amu. There are only two naturally occurring isotopes of gallium: 69Ga, with a mass of 69.0 amu, and 71Ga, with a mass of 71.0 amu. Calculate the natural abundance of the 69Ga isotope.

2. Magnesium contains three different isotopes: magnesium-24 with an abundance of 79% and a mass of 23.9850423 amu, magnesium-25 with an abundance of 10% and a mass of 24.9858374 amu, and magnesium-26 with a mass of 25.9825937 amu. Calculate the average atomic mass of a sample of magnesium.

10 Empirical Formula of an Oxide

EXPERIMENT

Empirical Formula of an Oxide

Goal

The goal of this experiment is to determine the *empirical formula* and *molecular formula* of magnesium oxide, produced by burning a known amount of Mg with oxygen from the air. Unfortunately, the oxidation of Mg to give MgO in the air also produces a small amount of magnesium nitride (Mg_2N_3), thinking that there is N_2 in the air. Adding water will get rid of the nitride by liberating ammonia (NH_3). You will determine the mass of oxygen that is present in the oxide by subtracting the mass of the product from the original mass of magnesium employed.

Materials

- ☐ 0.2 g of Mg ribbon
- ☐ Metallic wool
- ☐ Crucible with lid, ring stand with clay triangle

- ☐ Bunsen burner
- ☐ Crucible tongs
- ☐ A wire gauze

Background

Scientists often gain insight into the nature of chemicals by studying the properties of the compounds found in nature. Once they have extracted a compound from a natural resource, they try to identify its formula. In this experiment, we will identify the molecular formula of a chemical compound resulting from the reaction between Mg and O_2. This experiment is a well-known classic in college chemistry, implemented previously in numerous laboratory manuals [1–4].

Chemical formulas

The *empirical formula* of a compound shows the relative numbers of atoms of each element present in that compound. For example, the empirical formula of glucose, which is CH_2O, tells us that carbon, hydrogen, and oxygen atoms are present in the ratio 1:2:1. Differently, the *molecular formula* shows the actual numbers of atoms of each element in a molecule. The molecular formula for glucose, which is $C_6H_{12}O_6$, tells us that each glucose molecule consists of 6 carbon atoms, 12 hydrogen atoms, and 6 oxygen atoms.

To obtain the empirical formula, first, you need to convert the mass percentage of each type of atom in the compound into the relative number of atoms of that element. The simplest procedure is to imagine that we have a sample with a mass of exactly 100. g. In this case, the mass percentage composition gives the mass in grams of each element. Then we use the molar mass of each element to convert these masses into amounts in moles and go on to find the relative numbers of moles of each type of atom.

> **Example**
>
> *The mass percentage composition of a compound is:* 18.59% O, 37.25% S, and 44.16% F. Calculate its empirical formula.
>
> -
>
> **Answer**: the mass m of each atom in 100. g of the compound is:
>
> $$m(O) = 18.59\,g; \qquad m(S) = 37.25\,g; \qquad m(F) = 44.16\,g.$$

We convert the mass of each element into moles n:

$$n(O) = 1.16 \, mol; \qquad n(S) = 1.16 \, mol; \qquad n(F) = 2.32 \, mol.$$

We now divide each amount by the smallest amount (1.16):

$$O = 1; \qquad S = 1; \qquad F = 2.$$

The empirical formula is: OSF_2

Determining molecular formulas

The empirical formula of glucose is CH_2O. However, the empirical formula tells us only that the C, H, and O atoms are present in the sample in the ratio 1:2:1, not the number of each type of atom in a molecule. The molecular formula could be $C_2H_4O_2$ or $C_4H_8O_4$, or any other whole-number multiple of the empirical formula. To find the molecular formula of a compound, we need its molar mass.

Example

The molar mass of a compound is $104 \, g \cdot mol^{-1}$*, and its empirical formula is CH. Deduce the molecular formula of the compound.*

Answer: the molecular formula of the molecule is $n \cdot CH$, where n is an integer. Knowing that the molecular mass if $104 \, g \cdot mol^{-1}$ and the molecular mass of CH is $13 \, g \cdot mol^{-1}$, we can solve for n:

$$n \cdot 13 = 104$$

and $n = 8$. Hence the molecular formula is: C_8H_8.

The oxidation of Magnesium

Air is a mixture of different gases such as oxygen, nitrogen, water, or carbon dioxide. Oxygen (O_2) in the air, which is a diatomic molecule, is a very reactive substance and many elements will react with it forming what we call oxides. When Mg reacts with oxygen, magnesium oxide is formed:

$$2\,Mg \quad + \quad O_2 \quad \longrightarrow \quad 2\,MgO$$
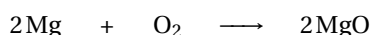
Nitrogen in the air N_2, also a diatomic molecule is normally inert, which means that it will hardly react. Still at very high-temperature metals can react with nitrogen producing what we call nitrides:

$$3\,Mg \quad + \quad N_2 \quad \longrightarrow \quad Mg_3N_2$$
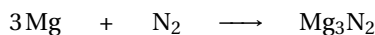
Using water and working still at high temperatures, we can eliminate Mg_3N_2 producing ammonia:
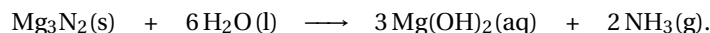
$$Mg_3N_2\,(s) \quad + \quad 6\,H_2O\,(l) \quad \longrightarrow \quad 3\,Mg(OH)_2\,(aq) \quad + \quad 2\,NH_3\,(g).$$

References

(1)   Ebbing, R. W.D. D., *Experiments in General Chemistry*; Houghton Mifflin Harcourt Publishing Company: 2004.

(2)   Russo, T., *Merrill Chemistry Lab Manual*; McGraw-Hill: 1998.

(3)   Fetzer-Gislason, P. R.D.W. S., *Lab Experiments in Introductory Chemistry*; Freeman: 2003.

(4)   Bull, W. B. E., *Laboratory Manual for College Chemistry*; SHarper and Row: 1980.

Procedure

Getting started

☐ *Step 1:* – Obtain a crucible with a lid, a clay triangle, and an iron ring attached to a ring stand.

☐ *Step 2:* – Place the covered crucible in the clay triangle on an iron ring attached to a ring stand. Adjust the height of the ring so that the bottom of the crucible will be in the hottest part of the flame. The correct arrangement of the equipment, crucible, and burner is shown in the figure (Right panel).

☐ *Step 3:* – Learn how to use the Bunsen burner ✍. Heat the covered crucible in the hottest part of the flame for about 5 min while keeping the lid ajar, making sure that the bottom of the crucible attains a red glow.

☐ *Step 4:* – Stop the burner and allow the crucible to cool down completely ⚠.

☐ *Step 5:* – Weight the covered crucible and record the mass of the covered crucible. Now you know the mass of the empty– and clean–crucible with lid.

Good Lab Practice

✍ Discharge the magnesium oxide in the disposal container.

✍ In the following video you can learn the correct use of a ☻ *Bunsen burner*.

Burning Magnesium

☐ *Step 6:* – Obtain 0.2 g of magnesium ribbon. Clean the surface of the metal with metallic wool until it shines.

☐ *Step 7:* – Cut the magnesium ribbon into tiny bits, and place them inside the crucible.

☐ *Step 8:* – Cover the crucible, obtain and record the mass again. Now you know the mass of the crucible+lid+Mg.

☐ *Step 9:* – Set the crucible on the clay triangle with the lid on and heat the crucible ✍ in the hottest part of the flame for another 5 min ⚠. Keep the lid closed.

☐ *Step 10:* – Using the crucible tongs, lift the lid carefully by a slight amount. The metal should glow brightly without flames. Continue until all Mg is burned and the product does not glow.

☐ *Step 11:* – Patiently cool down the crucible with a lid. The content should be white or slightly gray. At this point, add a few drops of water using a plastic pipet on the crucible content. You might notice a smell of ammonia at this point.

☐ *Step 12:* – Place the lid back onto the crucible, slightly ajar, and heat the crucible ⚠ in the hottest part of the flame for 15 more minutes.

☐ *Step 13:* – After that time, allow the covered crucible and its content to cool down. Obtain the mass of the covered crucible.

⚠ CAUTION!

⚠ Do not burn yourself with the crucible: it is hot.

⚠ Make sure the gas outlet is closed before leaving the lab.

Calculations

$\boxed{1}$ This is the mass of the empty and clean crucible with lid.

$\boxed{2}$ This is the mass of the clean crucible with lid and the Mg.

$\boxed{3}$ This is the mass of Mg added to the crucible: $\boxed{2}$–$\boxed{1}$

$\boxed{4}$ This is the moles of Mg (Atomic weight 24.305 $g \cdot mol^{-1}$):

$$n_{Mg} = \frac{\boxed{3}\,g}{24.305\,g \cdot mol^{-1}}$$

$\boxed{5}$ This is the mass of the clean crucible with lid and the product.

$\boxed{6}$ This is the mass of product: $\boxed{5}$–$\boxed{1}$

$\boxed{7}$ This is the mass of O in the product: $\boxed{6}$–$\boxed{3}$

$\boxed{8}$ This is the moles of O (Atomic weight 15.999 $g \cdot mol^{-1}$) in the product:

$$n_O = \frac{\boxed{7}\,g}{15.999\,g \cdot mol^{-1}}$$

Pre-lab Questions

Empirical Formula of an Oxide

1. Caffeine has a molar mass of $194.19\,g \cdot mol^{-1}$ and a mass percentage composition of 49.48% C, 5.19% H, 28.85% N, and 16.48% O. What is the molecular formula of caffeine?.

2. The percent composition of chloroform is 10.06% carbon, 0.85% hydrogen, and 89.09% chlorine. Determine the empirical formula.

3. In an experiment, $4.14g$ of Ni are burned to produce $4.88g$ of an oxide. Calculate the empirical formula of the compound. Mind that the indexes of a formula are always integer numbers.

4. When burning a pure metal to form an oxide, does the weight increase or decrease after the combustion?

Results
EXPERIMENT

Empirical Formula of an Oxide

1	Mass of empty crucible and lid (g)	
2	Mass of crucible and lid with Mg (g)	
3	Mass of Mg (g)	
4	Moles of Mg (mole)	
5	Mass of crucible and lid with MgO (g)	
6	Mass of MgO (g)	
7	Mass of O (g)	
8	Moles of O (moles)	

	Mg	O
Moles of (moles)		
Moles/smallest amount		
Empirical Formula		

Post-lab Questions

Empirical Formula of an Oxide

1. Write correctly balanced chemical equations for the following reactions:

    (a) Magnesium with molecular oxygen to produce magnesium oxide

    (b) Magnesium with molecular nitrogen to produce magnesium nitride

    (c) Magnesium nitride with water to produce magnesium hydroxide and ammonia

2. A 1.27-g sample of metallic copper was placed in a 26.32-g crucible and heated until all the copper reacted with the oxygen in the air to form an oxide. The crucible and product together were found to weigh 27.75 g. Calculate the empirical formula of the oxide and give the name.

3. Explain why did you have to shine the magnesium ribbon before the reaction.

EXPERIMENT

Absorption Spectrum of Cobalt(II) Chloride: Spectrochemistry

Goal

The goal of this experiment is to calculate the *concentration* of an unknown $CoCl_2$ solution using spectrochemistry and to get experience in the measurement of absorbance. To get started, you will need to find the *correct wavelength* of light to use in the measurements. This is the wavelength at which the maximum absorbance occurs. You will also practice dilutions, and you will *dilute* a concentrated cobalt solution to then measure the absorbance of the diluted solution. Finally, you will calculate backward the concentration in the original solution. The absorbance of a known cobalt solution will be measured before and after the dilutions.

Materials

- ☐ a $CoCl_2$ solution of known concentration
- ☐ 8 test tubes
- ☐ a spectrophotometer

- ☐ an unknown
- ☐ a 50 mL beaker
- ☐ a Mohr pipet (transfer pipet)

Background

When atoms are heated they emit light with very characteristic frequencies. At the same time, matter can also absorb discrete frequencies of light. The process of light absorption is exactly the opposite of light emission. During the absorption of light, an electron undergoes a transition from a lower-energy level to a higher-energy level, and the difference in energy corresponds to the energy of the light absorbed. As a result of the transition, a component of light–remember white light has components of different frequencies–with frequency v is absorbed and the other frequencies are transmitted across the material. This experiment is truly a classic in college chemistry, historically implemented in numerous lab manuals [1–4].

Transmittance and Absorbance

When light of a specific wavelength illuminates a solution of an absorbent substance, the intensity of the light diminishes as it passes through the solution. This is due to light absorption. If I_o is the intensity of the light that enters and I the intensity of the light that emerges from the solution, the transmittance is defined as:

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

A related quantity called absorbance, A, is defined as the negative logarithm of the transmittance

$$A = -log(T)$$

Spectrophotometers

Absorbance is measured with an instrument called a spectrophotometer. This instrument separates light into its component wavelengths and selectively measures the intensity of light of a given wavelength after it goes through a solution. All spectrophotometers have certain parts in common such as the source of light, a prism to select a particular wavelength, a device for holding the sample, and a detector that measures the intensity of the light.

Beer's Law

Beer's law states that the absorption A is directly related to the concentration c of a substance that absorbs light:

$$A = k_\lambda \cdot c$$

where k_λ is a constant. Because A is a dimensionless number and the unit of measurement for c is mol/L or M, it follows that the unit of measurement for k_λ is L/mol or M^{-1}. k is only a constant for a given substance at a particular wavelength, hence the subscript λ.

The determination of k_λ

The equation for Beer's law, $A = k \cdot c$, has the same form as the equation for a straight line, $y = mx + b$, where A is y, c is x and k_λ is the slope, m. Consequently, you should obtain a straight line when you plot the absorbances obtained at various concentrations against those concentrations. Moreover, the slope of that line will be given by k_λ and the line must pass through the origin because at zero concentration, zero absorbance.

Dilutions

Sometimes, in chemistry labs, one needs to obtain from a concentrated solution a more diluted one. To dilute a stock–more concentrated–solution to a desired–more diluted–concentration, a pipet is used to transfer the appropriate volume of stock solution to a volumetric flask with a given volume. Then the solvent is added to increase the volume of the solution to the volume of the flask. When diluting a certain volume V_1 of stock solution with concentration c_1 into a larger volume V_2, the formula that relates the stock solution concentration with the diluted solution c_2 is:

$$c_1 \cdot V_1 = c_2 \cdot V_2$$

Example

You need to prepare 250. mL of a diluted 1.25×10^{-3} M NaOH(aq) solution using a concentrated 0.0380 M NaOH(aq) stock solution. How much stock solution do you need?

- -

Answer: The original, stock solution has a concentration of $c_1 = 0.0380\,M$, whereas for the diluted solution should have a concentration $c_2 = 1.25 \times 10^{-3} M$. We need a volume $V_2 = 250.\,mL$. We solve for V_1 to know how much of the stock solution we need to take and later dilute with the solvent.

$$c_1 \cdot V_1 = c_2 \cdot V_2 \qquad V_1 = \frac{c_2 \cdot V_2}{c_1}$$

$$V_1 = \frac{1.25 \times 10^{-3} M \cdot 250.\,mL}{0.0380 M}$$

and this gives $V_1 = 8.22\,mL$.

References

(1)   Murov, S., *Experiments in General Chemistry*; Cengage: 2013.

(2)   Sawyer, R. B.J.B. D., *Laboratory Inquiry in Chemistry*; Cengage: 2009.

(3)   Gunnerson, C. C. K., *Exploring General Chemistry in the Laboratory*; Morton: 2017.

(4)   Rossi, E. S.W.W. R., *Chemical Principles in the Laboratory*; Cengage: 2014.

Procedure

Part A: Calculating the maximum wavelength λ_{max}

☐ *Step 1:* – Make sure that there is no cuvette in the spectrophotometer. Close the lid and start the device.

☐ *Step 2:* – Obtain 7 test tubes.

☐ *Step 3:* – Mark each test tube with one of a series of identification numbers running from #1 to #7.

☐ *Step 4:* – Get 30 mL of the stock $CoCl_2$ solution in a 50 mL beaker to prepare a series of dilutions from this solution. Record the concentration (Molarity) written on the bottle in the Results sheet.

☐ *Step 5:* – Use Mohr pipets to make additions of $CoCl_2$ and distilled water as shown in the table below. Make sure your instructor shows you how to use this pipet.

Test tube #	V_{CoCl_2} (mL)	$V_{Water(mL)}$
#1	5.0	0.0
#2	4.0	1.0
#3	3.5	1.5
#4	3.0	2.0
#5	2.5	2.5
#6	2.0	3.0
#7	1.0	4.0

☐ *Step 6:* – Gently swirl the solutions from side to side to make sure they are mixed.

☐ *Step 7:* – Set the wavelength at 400 nm.

☐ *Step 8:* – Fill a cuvette with water, make sure the walls of the cuvette are clean and dry, and make the absorbance of this sample zero–this is blank.

☐ *Step 9:* – Fill another cuvette with the solution in the first test tube. Ideally, you will rinse the cuvette first with 1 mL of the solution to be used. Place the cuvette in the holder and read the absorbance for 400 nm wavelength. You do not need to touch anything to measure. Just record your result. Mind not disposing of this solution after the measurement.

☐ *Step 10:* – Repeat the previous 3 steps, but this time for 425 nm, then for 475 nm, and so on. Until reaching 600 nm. You need to redo the blank every time you change the wavelength. In other words, you need to change the wavelength, place the water cuvette, press zero, add the sample, and record.

☐ *Step 11:* – Graph Absorbance vs. wavelength.

☐ *Step 12:* – From the plot, select the wavelength at which the absorbance is largest.

☐ *Step 13:* – **Use this wavelength for all subsequent measurements.**

Good Lab Practice

✍ Mind not to use your finger as tube stopper. ✍

Part B: Constructing the calibration curve

☐ *Step 14:* – Select the maximum wavelength obtained in the previous step in the spectrophotometer, and do the blank for this wavelength. After this point, there is no need to do the blank anymore.

☐ *Step 15:* – Measure again the absorbance of the first test tube and record the results.

☐ *Step 16:* – Measure the absorbance of the remaining tubes and record your results.

☐ *Step 17:* – Plot Absorbance vs. concentration with Absorbance on the Y and concentration on the X. To do that, follow the steps to fill in the values on the Results table.

☐ *Step 18:* – Calculate the slope of the graph by using the formula:

$$k = \frac{\sum c \cdot A}{\sum c^2}$$

☐ *Step 19:* – Show the results to your instructor to ensure your plot is correct.

Part C: Measuring the unknown

☐ *Step 20:* – Make sure you use the maximum wavelength obtained in Part A.

☐ *Step 21:* – Using a new test tube, add 2 mL of the unknown and 2 mL of water (we will call this V_{water}) and measure the absorbance. If the absorbance is not within the range of your previous results, change relative quantities of water and unknown.

☐ *Step 22:* – Knowing k_λ from the previous part of the experiment, solve for the concentration of the diluted solution $c_{Diluted}$:

$$c_{Diluted} = \frac{A}{k_\lambda}$$

☐ *Step 23:* – Now go back from the concentration of the diluted solution $c_{Diluted}$ to calculate the concentration of the unknown using the formula:

$$c_{Unknown} \cdot V_{unknown} = c_{Diluted} \cdot V_{Diluted}$$

☐ *Step 24:* – Make sure you stop the spectrophotometer and remove the cuvette.

Calculations

⓪ This is the wavelength that gives a maximum absorbance.

① This is the volume of cobalt solution added to the tube.

② This is the volume of water added to the tube.

③ This is the total volume and should always be 5mL: ① + ②

④ This is the concentration of cobalt after dilutions in each tube, $c_{diluted}$:

$$c_{diluted} = \frac{c_{CoCl2} \cdot ①}{③}$$

⑤ The measured absorbance

⑥ The product of the concentration and the absorbance for each measurement ⑤ × ④

⑦ The square of each diluted concentration ④ ^{2}

⑧ The sum ($\sum$) of all the values in column ⑥ and the sum for all the values for column ⑦ respectively.

⑨ Here you calculate the slope of the A vs. c plot by doing:

$$k = \frac{\sum c \cdot A}{\sum c^2}$$

⑩ The volume of unknown you used.

⑪ The volume of water you added.

⑫ The absorbance you measured.

⑬ The concentration of cobalt in the sample you measured that is diluted is:

$$c_{Co,diluted} = ⑫ / ⑨$$

⑭ The concentration of cobalt in the real original sample is:

$$c_{Unknown} = \frac{⑬ \cdot \left(⑩ + ⑪ \right)}{⑩}$$

Pre-lab Questions

Absorption Spectrum of Cobalt(II) Chloride: Spectrochemistry

1. The absorbance of a colored substance in a colorless liquid is measured at each of a series of wavelengths, and the data is given below:

λ(nm)	325	350	375	400	425	450	475	500	525
A	0.016	0.144	0.341	0.578	0.681	0.558	0.281	0.092	0.031

   (a) Plot A vs. λ in the graph below.

   (b) Calculate the λ value that gives a maximum A.
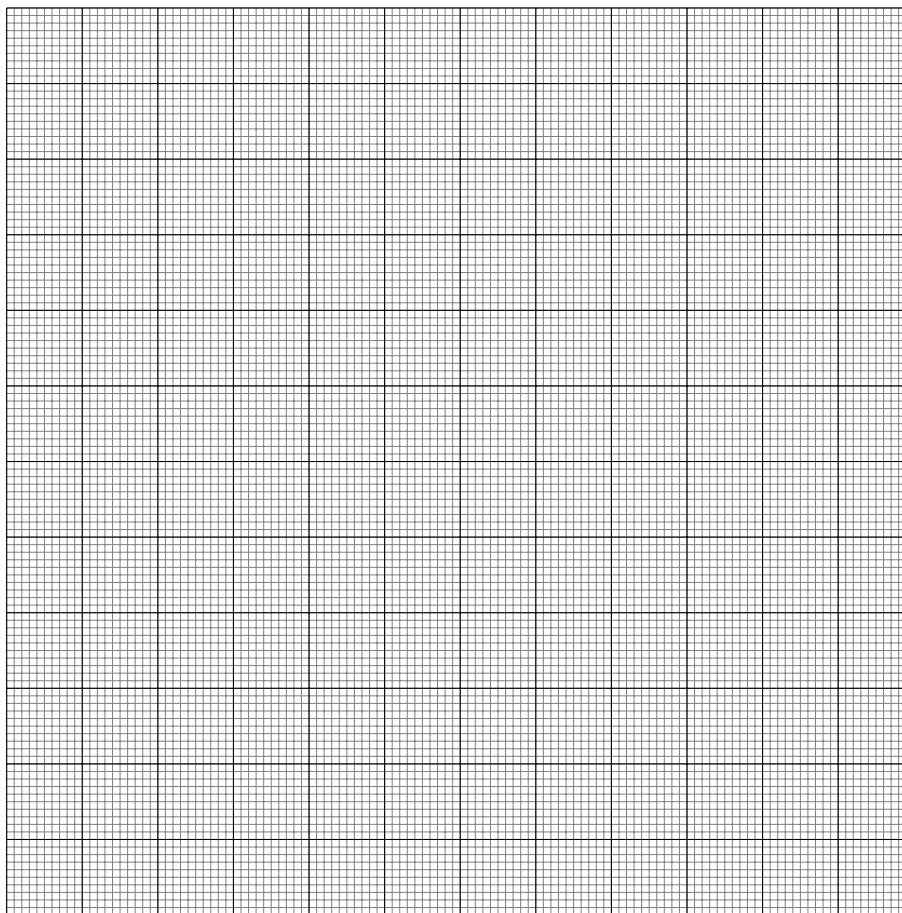
$$\lambda(A_{max}) \quad = \underline{\hspace{5cm}}$$

2. A set of absorbances, A, for different concentrations, c, are given below.

   (a) Plot A vs. c in the graph below.

   (b) Compute the slope of the graph by using the formula:

$$k = \frac{\sum c \cdot A}{\sum c^2}$$

c (M)	A	$c \cdot A$ (M)	c^2 (M^2)
0.0120	0.681		
0.00960	0.540		
0.00720	0.389		
0.00480	0.270		
0.00240	0.133		
Sum			
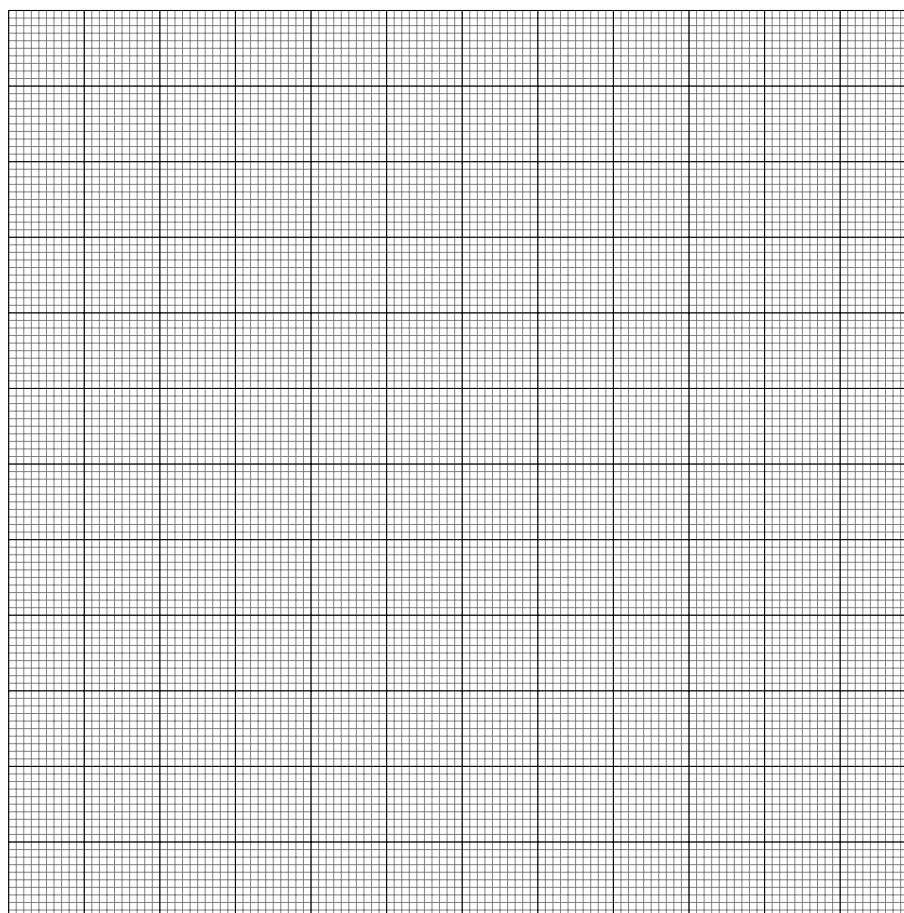
$$k \ (M^{-1}) = \underline{\hspace{5cm}}$$

3. After calculating k, now write down the formula for absorbance in the form: $A = k \cdot c$

Results
EXPERIMENT

Absorption Spectrum of Cobalt(II) Chloride: Spectrochemistry

Calculating the wavelength

λ (nm)	400	425	475	500	525	550	575	600
A								

(0)   Max Abs. λ (nm)=_____ c_{CoCl_2} (M)=_____

Constructing the calibration curve

Test tube #	V_{CoCl_2} (mL)	V_{water} (mL)	V_{Total} (mL)	c_{CoCl_2} (M)	Absorbance A	$A \cdot c_{CoCl_2}$	$c_{CoCl_2}^2$
	①	②	③	④	⑤	⑥	⑦
#1	5.0	0.0					
#2	4.0	1.0					
#3	3.5	1.5					
#4	3.0	2.0					
#5	2.5	2.5					
#6	2.0	3.0					
#7	1.0	4.0					

⑧ Sum (Σ)=

⑨ k_λ=

Measuring the unknown

Unknown Number =_____

Measurement of the unknown				
$V_{unknown}$ (mL) ⑩	V_{water} (mL) ⑪	Absorbance A ⑫	$c_{diluted}$ (M) ⑬	$c_{unknown}$ (M) ⑭
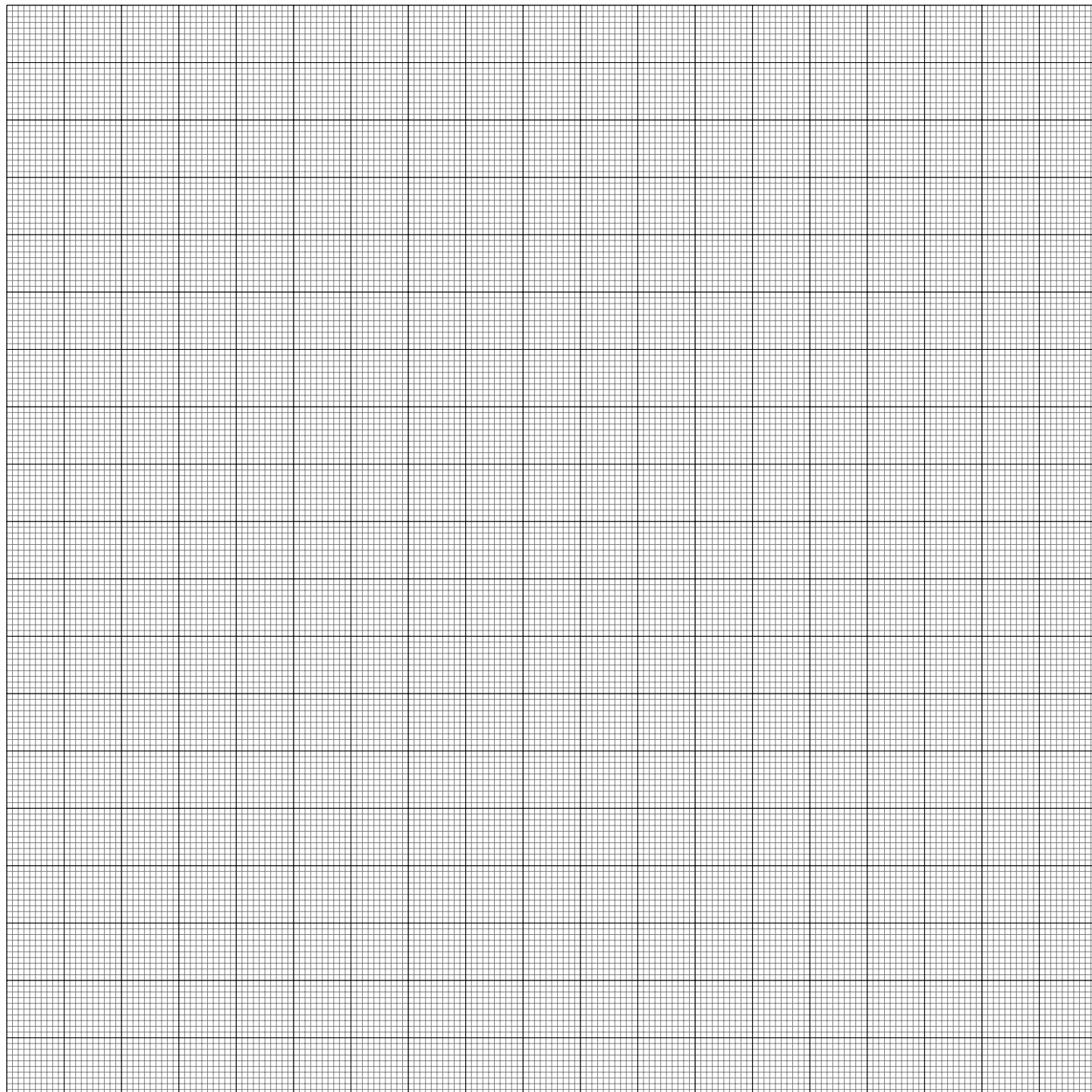
Figure 4: λ (X axis) vs. A (Y axis)
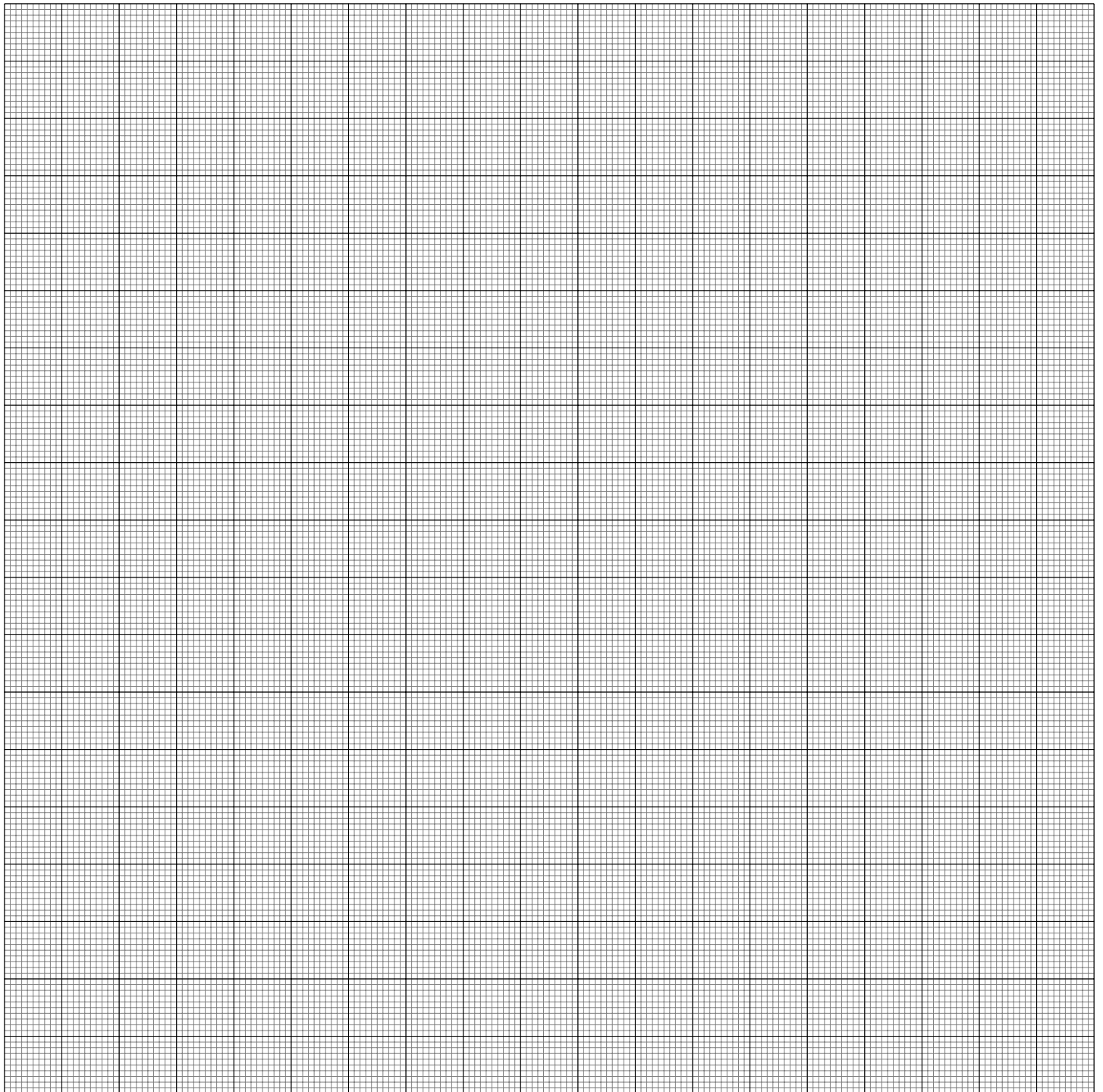
Figure 5: c (X axis) vs. A (Y axis)

Post-lab Questions

Absorption Spectrum of Cobalt(II) Chloride: Spectrochemistry

1. Write a brief procedure outlining how would you prepare 250. mL of a 0.150 M solution of $CoCl_2$ from solid $CoCl_2$ and distilled water.

2. Using the solution from the previous question, how would you prepare 100mL of a 0.060M diluted $CoCl_2$ solution.

3. What is the purpose of obtaining the wavelength of maximum absorbance?

EXPERIMENT

Hydrates and Their Thermal Decomposition

Goal

The goal of this experiment is to study the thermal decomposition of a hydrate–barium chloride dihydrate–and to compare this reaction with the thermal decomposition of a few other hydrates. The thermal decomposition of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) produces water, whereas other oxides often generate hydrochloric acid.

Materials

- □ Crucible and lid.
- □ Crucible tongs and wire gauze.
- □ about 1.5 g of $BaCl_2 \cdot 2H_2O$ (s).

- □ small test tubes.
- □ litmus paper.
- □ $CrCl_3 \cdot 6H_2O$ (s) and $CoCl_2 \cdot 6H_2O$ (s).

Background

The amount of product generated in a chemical decomposition can be predicted using stoichiometric calculations. Knowing the initial mass of the reactant the amount (mass) of products and more importantly, the mass increase or decrease in the reaction can be estimated. The thermal decomposition of $BaCl_2 \cdot 2H_2O$–an extensively studied reaction in the college laboratory literature [1–4]–generates $BaCl_2$ (s) and H_2O (g). If you start with a certain mass of $BaCl_2 \cdot 2H_2O$ the weight of the final product should be lower, as the hydrate loses water molecules during the reaction.

Basic reaction Stoichiometry

When studying chemical reactions, oftentimes one needs to predict how much product can be produced from a given amount of reactants. To obtain this type of information we use *reaction stoichiometry*, and the key to employing reaction stoichiometry is a balanced chemical equation. The coefficients from the balanced chemical equation, known as stoichiometric coefficients, are needed to relate one substance with another.

Example

What is the number of moles of H_2O that can be produced from 2.0 mol $BaCl_2 \cdot 2H_2O$ in the reaction:

$$BaCl_2 \cdot 2H_2O\,(s) \quad \longrightarrow \quad BaCl_2\,(s) \quad + \quad H_2O\,(g)$$

Answer: the first step would be to balance the reaction:

$$BaCl_2 \cdot 2H_2O\,(s) \quad \longrightarrow \quad BaCl_2\,(s) \quad + \quad \mathbf{2}H_2O\,(g)$$

The reaction produces two water molecules. We will relate the number of H_2O moles with the given number of $BaCl_2 \cdot 2H_2O$

moles using a conversion factor from the mole–to–mole ratio

$$n(H_2O) = 2.0 \, mol \, BaCl_2 \cdot 2H_2O \times \frac{2 \, mol \, H_2O}{3 \, mol \, BaCl_2 \cdot 2H_2O} = 1.3 \, mol \, H_2O$$

Mass-to-mass predictions

To calculate how much product can be produced from a given mass of a reactant, we need first to convert the grams of reactant into moles, using its molar mass, to then use the mole ratio from the balanced equation. Finally, we need to convert the moles of product formed, back into mass units using the molar mass.

Example

Sulfur trioxide $BaCl_2 \cdot 2H_2O$ (MW 244.266 $g \cdot mol^{-1}$) decomposes to produce $BaCl_2$ (MW 208.236 $g \cdot mol^{-1}$) and H_2O (MW 18.015 $g \cdot mol^{-1}$):

$$BaCl_2 \cdot 2H_2O \, (s) \quad \longrightarrow \quad BaCl_2 \, (s) \quad + \quad 2H_2O \, (g)$$

What mass of $BaCl_2$ will be produced from 3.00 g of $BaCl_2 \cdot 2H_2O$?

Answer: knowing that the equation is already balanced, we will first convert the grams of $BaCl_2 \cdot 2H_2O$ into moles to then relate the $BaCl_2 \cdot 2H_2O$ with the $BaCl_2$ moles produced. Finally, we will convert the $BaCl_2$ moles back into grams:

$$m(BaCl_2) = 3.00 \, g \, BaCl_2 \cdot 2H_2O \times \frac{1 \, mol \, BaCl_2 \cdot 2H_2O}{244.266 \, g \, BaCl_2 \cdot 2H_2O} \times \frac{1 \, mol \, BaCl_2}{1 \, mol \, BaCl_2 \cdot 2H_2O} \times \frac{208.236 \, g \, BaCl_2}{1 \, mol \, BaCl_2} = 2.56 \, g \, BaCl_2$$

Hydrates

Some compounds in contact with the moisture of air form crystals that incorporate water molecules. These compounds are called *hydrates.* Hydrates are named by first giving the name of the compound, followed by the word *hydrate* with a Greek prefix (mono, di, tri, etc.) indicating how many molecules of water are found in each formula unit. For example, the name of $CuSO_4 \cdot 5H_2O$ is copper(II) sulfate pentahydrate. The quantity of water will not change as long as the temperature (and pressure) is not changed. A substantial increase in temperature by heating the chemical, however, will cause the loss of water. When we want to emphasize that a compound has lost its water of hydration, we call it *anhydrous.*

Example

Name $BaCl_2 \cdot 2H_2O$ and give the formula for Sodium carbonate pentahydrate.

Answer: $BaCl_2 \cdot 2H_2O$ is named barium chloride dihydrate, whereas the formula for sodium carbonate pentahydrate is $Na_2CO_3 \cdot 5H_2O$.

Water composition of a hydrate

Different hydrates have different water compositions. For example $BaCl_2 \cdot 2H_2O$ contains two water molecules, whereas $CuSO_4 \cdot 5H_2O$ contains five water molecules. At the same time, each hydrate will have a different mass % of water, depending on its water composition. $BaCl_2 \cdot 2H_2O$ is 15% water whereas $CuSO_4 \cdot 5H_2O$ is 36% water.

Example

Calculate the mass % of water in $BaCl_2 \cdot 2H_2O$ (MW 244.266 $g \cdot mol^{-1}$) and in $CuSO_4 \cdot 5H_2O$ (MW 250 $g \cdot mol^{-1}$).

Answer: $BaCl_2 \cdot 2H_2O$ has two water molecules and a molecular weight of 244.266 $g \cdot mol^{-1}$, and hence the mass % of water will be $\frac{2 \times 18.015 \, g \, H_2O}{244.266 \, g \, BaCl_2 \cdot 2H_2O} \times 100 = 14.750 \, \%$ of water. $CuSO_4 \cdot 5H_2O$ has five water molecules and a molecular weight of 249.681 $g \cdot mol^{-1}$, and hence the mass % of water will be $\frac{5 \times 18.015 \, g \, H_2O}{249.681 \, g \, CuSO_4 \cdot 5H_2O} \times 100 = 36.076\%$ water.

Litmus paper

Some hydrates produce acid when decomposing. Litmus paper is a quick test to identify whether a hydrate generates an acid. Litmus paper is a standard acid-base indicator. There are two variants for litmus paper. You can find red litmus paper and blue litmus paper. Blue litmus paper turns pink under acidic conditions and red litmus paper turns blue under basic or alkaline conditions.

Example

A solution is tested with red litmus and the paper turned blue. Indicate whether the solution is acidic or basic.

- -

Answer: Acids turn blue litmus into pink and bases turn red litmus into blue. In this case, as the red indicator turned blue, that means that the solution was a base.

Solution	Red litmus paper	Blue litmus paper
Acidic	Red	Red
Basic	Blue	Blue

Heating hydrates

As hydrates contain water, if we warn them they will release this water and will reduce its mass. A way to calculate the amount of water in a hydrate is to warm up a sample and measure the reduction in mass. This mass reduction corresponds to the mass of water in the compound.

Example

A mass of 1g of $BaCl_2 \cdot xH_2O$ (s) becomes 0.9 after heating the hydrate for a long enough time. Calculate the percentage of mass in the hydrate.

- -

Answer: The mass of water if 0.1g, as 1g of hydrate weighs 0.9g after the thermal decomposition. Therefore, the chemical has 10% of mass.

References

(1) Ebbing, R. W.D. D., *Experiments in General Chemistry*; Houghton Mifflin Harcourt Publishing Company: 2004.

(2) Bishop, K. W.C.B. M., *Experiments in General Chemistry*; Saunders College Publishing: 1992.

(3) Corwin, C., *Laboratory Experiments*; Prentice-Hall: 1985.

(4) Beran, J. A., *Laboratory manual for principles of general chemistry*; John Wiley and Sons: 2010.

Procedure

Getting started

☐ *Step 1:* – Place a clean, covered crucible in a clay triangle on an iron ring attached to a ring stand. Adjust the height of the ring so that the bottom of the crucible will be in the hottest part of the flame. The correct arrangement of the equipment, crucible, and burner is shown in the figure (Right panel).

☐ *Step 2:* – Learn how to use the Bunsen burner ✍. Heat the covered crucible in the hottest part of the flame for about 5 min, making sure that the bottom of the crucible attains a red glow.

☐ *Step 3:* – Stop the burner and allow the crucible to cool down completely ⚠.

☐ *Step 4:* – Weight the covered crucible and record the mass of the covered crucible.

> **Good Lab Practice**
>
> ✍ Learn the correct use of a ☝ *Bunsen burner*.
>
> ✍ Learn how to ☝ *warm a test tube* on the flame.
>
> ✍ Make sure the gas outlet is closed before leaving the lab.

Decomposition of $BaCl_2 \cdot 2H_2O$

☐ *Step 5:* – Weight about 1.5 g of $BaCl_2 \cdot 2H_2O$.

☐ *Step 6:* – Add the $BaCl_2 \cdot 2H_2O$ sample onto the crucible and cover the crucible again. Weight the covered crucible with the $BaCl_2 \cdot 2H_2O$ and record the exact mass in the results sheet.

☐ *Step 7:* – Heat the crucible in the hottest part of the flame for about 15 min. The bottom of the crucible should be red hot during this time.

☐ *Step 8:* – When the crucible is cool, weigh and record the mass of the product.

Hydrates producing acid

☐ *Step 9:* – Place a pea-sized portion of $CrCl_3 \cdot 6H_2O$ in a test tube.

☐ *Step 10:* – Using the Bunsen Burner ⚠ and with a test tube clamp, hold the test tube at an angle of about 45° while swinging it above the hottest part of the flame.

☐ *Step 11:* – When steam forms test with blue litmus paper the liquid drops that condensate near the opening of the test tube.

☐ *Step 12:* – Repeat the previous 3 steps using $BaCl_2 \cdot 2H_2O$ and $CoCl_2 \cdot 6H_2O$ instead.

> ⚠ **CAUTION!**
>
> ⚠ Do not touch the crucible or the iron ring during this experiment as it should be hot.
>
> ⚠ Be aware that hot glassware look the same as when cold.

Calculations

(1) Record the mass of the empty crucible with the lid. Remember to weight the crucible in the balance only when completely cool.

(2) Record the mass of the empty crucible with the lid with $BaCl_2 \cdot 2H_2O$.

(3) The mass of $BaCl_2 \cdot 2H_2O$ added to the crucible should be:

$$\text{Mass of } BaCl_2 \cdot 2H_2O = \boxed{2} - \boxed{1}$$

(4) After you heat the crucible with $BaCl_2 \cdot 2H_2O$ a product will form. Weight the crucible and lid with the final product inside.

(5) You should calculate the mass of product by doing:

$$\text{Mass Product} = \boxed{4} - \boxed{1}$$

(6) Calculate the mass % of water in the hydrate:

$$\frac{(\text{Mass } BaCl_2 \cdot 2H_2O) - (\text{Mass Product})}{(\text{Mass of } BaCl_2 \cdot 2H_2O)} \times 100 = \frac{\boxed{3} - \boxed{5}}{\boxed{3}} \times 100$$

(7) Calculate the theoretical mass % of water in the hydrate

Hydrates and Their Thermal Decomposition

1. Name or give the formula of the following compounds:

   Magnesium sulfate _____

   $MgSO_4 \cdot H_2O$ _____

   Barium chloride _____

   $BaCl_2 \cdot 2\,H_2O$ _____

   Chromium(III) chloride _____

   $CoCl_2 \cdot 6\,H_2O$ _____

   Nickel(II) sulfate heptahydrate _____

2. The thermal decomposition of 2.00 g of $MgSO_4 \cdot H_2O$ (MW 138.383 $g \cdot mol^{-1}$) produces 1.7 g of a$MgSO_4$ (MW 120.366 $g \cdot mol^{-1}$).

$$MgSO_4 \cdot H_2O\,(s) \xrightarrow{\Delta} MgSO_4\,(s) + H_2O\,(g)$$
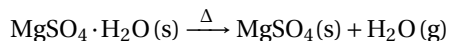
   (a) Calculate the mass of water in the compound.

   (b) Calculate the water % mass in the compound.

3. Calculate the theoretycal mass % of water of the hydrate: $Li_2SO_4 \cdot H_2O\,(s)$.

4. A solution is tested with blue litmus and the paper turns red. Indicate whether the solution is acidic or basic.

Results
EXPERIMENT

Hydrates and Their Thermal Decomposition

Decomposition of $BaCl_2 \cdot 2H_2O$

① Mass of empty crucible and lid (g)	
② Mass of crucible and lid with $BaCl_2 \cdot 2H_2O$ (g)	
③ Mass of $BaCl_2 \cdot 2H_2O$ (g)	
④ Mass of crucible, lid and product (g)	
⑤ Mass of dry product (g)	
⑥ Mass % of water	
⑦ Theoretical mass % of water in $BaCl_2 \cdot 2H_2O$	

Hydrates producing acid

	Litmus Color Before	Litmus Color After	Acid produced?
$BaCl_2 \cdot 2H_2O$			
$CrCl_3 \cdot 6H_2O$			
$CoCl_2 \cdot 6H_2O$			

STUDENT INFO

Name: Date:

Post-lab Questions

Hydrates and Their Thermal Decomposition

1. Name or give the formula of the following compounds:

   Zinc sulfate   _____

   $ZnSO_4 \cdot 7H_2O$ _____

   Iron(II) sulfate   _____

   $FeSO_4 \cdot 7H_2O$ _____

   Sodium carbonate   _____

   $Na_2CO_3 \cdot 10H_2O$ _____

2. 3.0 grams of an unknown hydrate is thermally decomposed giving 2.0 grams of the product.

   (a) Calculate the mass of water in the hydrate

   (b) Calculate the mass % of water in the hydrate.

Ionic Reactions in Aqueous Solutions: Precipitation and acid-base reactions

Goal

The goal of this experiment is to carry out a set of simple acid-base and precipitation reactions in aqueous solutions. On one hand, you will carry precipitation reactions and you will put into practice the *solubility rules*. You will have to observe the final product of the reaction and identify it as *soluble* or *insoluble*. On the other hand, you will examine some simple *acid-base reactions*, identify the acidic/basic character of each chemical and figure out whether there is heat involved in the acid-base reaction.

Materials

- ☐ Red or blue litmus paper.
- ☐ 4 small test tubes.
- ☐ solutions of:
  NH_4NO_3 (aq), HCl (aq), CH_3COOH (aq),
- ☐ solutions of:
  $NaOH$ (aq), Na_2CO_3 (aq) and NH_3 (aq)

- ☐ solutions of:
  $NaCl$ (aq), $NaBr$ (aq), Na_2SO_4 (aq),
  and KCl (aq)

- ☐ solutions of:
  $Ba(NO_3)_2$ (aq), $AgNO_3$ (aq), $Pb(NO_3)_2$ (aq),
  and $Ni(NO_3)_2$ (aq)

Background

Many chemical reactions take place in aqueous solution, and most of these reactions involve ions. Let's consider as an example what happens when mixing a colorless solution of silver nitrate ($AgNO_3$) with a colorless solution of sodium chloride ($NaCl$). The solution of silver nitrate contains Ag^+ cations and NO_3^- anions, whereas the solution of sodium chloride contains Na^+ cations and Cl^- anions. When we mix these two aqueous solutions, a white precipitate ($AgCl$) forms immediately due to the ion exchange process.

$$AgNO_3\,(aq) \quad + \quad NaCl\,(aq) \quad \longrightarrow \quad AgCl\,(s) \quad + \quad NaNO_3\,(q)$$
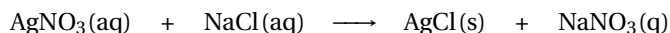
On the other hand, acid-base reactions are an important type of aqueous reaction. Acids–substances with a sharp or sour taste–and bases–chemicals with a soapy feel– react through a neutralization reaction. An example would be:

$$HCl\,(aq) \quad + \quad NaOH\,(aq) \quad \longrightarrow \quad NaCl\,(aq) \quad + \quad H_2O\,(l)$$
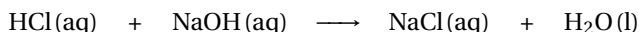
This experiment addresses these two important types of chemical reactions, studying several classic precipitation and acid-base reactions. Similar experiments have been performed in the literature [1, 2], and some of the reagents of this experiment are adapted from [3].

Weak and strong electrolytes

Electrolytes are chemicals that when placed in water break or dissociate into ions with positive and negative charges. An example of an electrolyte would be NaCl, which is a solid. When dissolved in water it produces Na^+ and Cl^-. On the other hand, non-electrolytes are chemicals that when dissolved in the water stay in their molecular form. An example of this is ethanol, CH_3CH_2OH, that you can find in wine. Once dissolved in water it stays as a molecule and does not produce positive and negative ions. Electrolytes can be classified as strong or weak electrolytes, depending on their degree of dissociation in water. Strong electrolytes dissociate completely producing large amounts of ions, whereas weak electrolytes dissociate only partially to produce just a few ions.

Precipitation reactions

Some ionic compounds are soluble in water whereas others are not. In a precipitation reaction, two electrolyte solutions are mixed to produce an insoluble solid called *precipitate*. When an insoluble substance is formed in water, it immediately precipitates and separates from the solution in solid form.

Table 2 Solubility rules	
Ions that form *soluble* compounds...	... **except** when combined with
Group I ions (Na^+, Li^+, K^+, etc)	no exceptions
Ammonium (NH_4^+)	no exceptions
Nitrate (NO_3^-)	no exceptions
Acetate (CH_3COO^-)	no exceptions
Hydrogen carbonate (HCO_3^-)	no exceptions
Chlorate (ClO_3^-)	no exceptions
Halides (F^-, Cl^-, Br^-)	Pb^{2+}, Ag^+ and Hg_2^{2+}
Sulfate (SO_4^{2-})	Ag^+, Ca^{2+}, Sr^{2+}, Ba^{2+}, Hg_2^{2+} and Pb^{2+}
Ions that form *insoluble* compounds...	... **except** when combined with
Carbonate (CO_3^{2-})	group I ions (Na^+, Li^+, K^+, etc) or ammonium (NH_4^+)
Chromate (CrO_4^{2-})	group I ions (Na^+, Li^+, K^+, etc) or Ca^{2+}, Mg^{2+} or ammonium (NH_4^+)
Phosphate (PO_4^{3-})	group I ions (Na^+, Li^+, K^+, etc) or ammonium (NH_4^+)
Sulfide (S^{2-})	group I ions (Na^+, Li^+, K^+, etc) or ammonium (NH_4^+)
Hydroxide (OH^-)	group I ions (Na^+, Li^+, K^+, etc) or Ca^{2+}, Mg^{2+}, Sr^{2+} or ammonium (NH_4^+)

Ionic and Net ionic equations

The ionic equation for a precipitation reaction shows all the species as they exist in the solution. Because dissolved ionic compounds exist as separate aqueous ions, in an ionic equation the ions should be shown separately. Some of these ions appear as both reactants and products. This means that they play no role in the reaction: they are spectator ions. In the ionic equation, you can simplify the chemical equation by canceling the spectators out on each side of the arrow.

Example

> *Write the net ionic equation for the reaction between aqueous solutions of silver nitrate ($AgNO_3$) and potassium chromate (K_2CrO_4) to give a precipitate of silver chromate (Ag_2CrO_4) and a solution of potassium nitrate (KNO_3):*
>
> $$AgNO_3(aq) \; + \; K_2CrO_4(aq) \; \longrightarrow \; Ag_2CrO_4(s) \; + \; KNO_3(aq)$$
>
> -
>
> **Answer**: the complete ionic equation, which shows all the dissolved ions, both those that react and the spectator ions, is:
>
> $$Ag^+(aq) \; + \; NO_3{}^-(aq) \; + \; 2K^+(aq) \; + \; CrO_4{}^{2-}(aq) \; \longrightarrow \; Ag_2CrO_4(s) \; + \; K^+(aq) \; + \; NO_3{}^-(aq)$$
>
> Now cancel the spectator ions K^+ and $NO_3{}^-$
>
> $$2Ag^+(aq) \; + \; \cancel{2NO_3{}^-(aq)} \; + \; \cancel{2K^+(aq)} \; + \; CrO_4{}^{2-}(aq) \; \longrightarrow \; Ag_2CrO_4(s) \; + \; \cancel{2K^+(aq)} \; + \; \cancel{2NO_3{}^-(aq)}$$
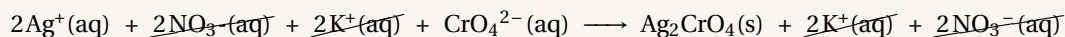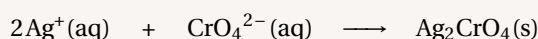>
> and obtain the net ionic equation:
>
> $$2Ag^+(aq) \; + \; CrO_4{}^{2-}(aq) \; \longrightarrow \; Ag_2CrO_4(s)$$
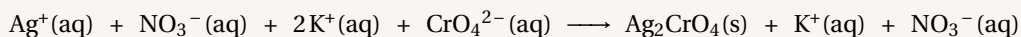
Acids and bases

Acids are chemicals that produce hydrogen ions (H^+, also known as protons) in water. Bases, on the other hand, accept hydrogen ions, producing hydroxide ions (OH^-) in water. Acids and bases change the color of certain chemicals called indicators, and litmus is a well-known acid-base indicator. Aqueous solutions of acids turn litmus red, whereas aqueous solutions of bases turn it blue. Acids and bases can be classified as strong or weak according to the extent to which they dissociate in solution. This way, a strong acid is completely dissociated in solution, whereas a weak acid is only slightly dissociated. The same can be applied to bases.

Table 3 Weak and strong acids	
Strongs acids	*Strongs* bases
Hydrobromic acid (HBr)	Group I hydroxides (NaOH, KOH, etc.)
Hydrochloric acid, (HCl)	Alkaline earth metal hydroxides ($Ba(OH)_2$, $Mg(OH)_2$, etc.)
Hydroiodic acid, (HI)	Group I and Group II oxides (Na_2O, BaO, etc.)
Nitric acid (HNO_3)	
Perchloric acid ($HClO_4$)	
Chloric acid ($HClO_3$)	
Sulfuric acid (H_2SO_4)	
Weak acids	*Weak* bases
$HF(aq)$, $CH_3COOH(aq)$, $H_2SO_3(aq)$	$NH_3(aq)$

Neutralization

A neutralization reaction is a reaction between a strong acid and a strong base to give salt and water:

$$Acid(aq) \; + \; Base(aq) \; \longrightarrow \; Salt(aq) \; + \; H_2O(l)$$
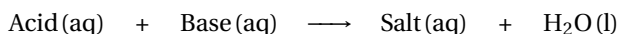
When we write the net ionic equation for the neutralization of a weak acid or a weak base, we use the molecular form of the weak acid or base and not its ionic form, because molecules are the dominant species in solution.

Litmus paper

Litmus paper is a quick test to identify whether a solution is acidic or basic. There are two variants for litmus paper. You can find red litmus paper and blue litmus paper. Blue litmus paper turns pink under acidic conditions whereas red litmus paper turns blue under basic conditions.

Solution	Red litmus paper	Blue litmus paper
Acidic	Red	Red
Basic	Blue	Blue

References

(1)   Fetzer-Gislason, P. R.D.W. S., *Lab Experiments in Introductory Chemistry*; Freeman: 2003.

(2)   Murov, S., *Experiments in General Chemistry*; Cengage: 2013.

(3)   Ebbing, R. W.D. D., *Experiments in General Chemistry*; Houghton Mifflin Harcourt Publishing Company: 2004.

Procedure

Getting started

☐ *Step 1:* – Obtain the material you need (litmus paper, test tubes, reactants).

☐ *Step 2:* – Obtain instructions on how to use the centrifuges from the lab instructor to separate some of the insoluble compounds. You can learn more in the video link early in the chapter.

☐ *Step 3:* – Prepare solutions of NaCl and KCl by weighting 0.24g of KCl and 0.12g of NaCl. Dissolve each salt in 25 mL of distilled water. Label the solutions.

> **Good Lab Practice**
>
> ✍ Learn the correct use of a ❂ *centrifuge*.
>
> ✍ Discharge the contents of each test tube as directed by your lab instructor.

Part A: Precipitation reactions

☐ *Step 4:* – Arrange in the following order the set of reactants Type A: $NaCl(aq)$, $NaBr(aq)$, $Na_2SO_4(aq)$ and $KCl(aq)$

☐ *Step 5:* – Arrange in the following order the set of reactants Type B: $Ba(NO_3)_2(aq)$, $AgNO_3(aq)$, $Pb(NO_3)_2(aq)$ and $Ni(NO_3)_2(aq)$

☐ *Step 6:* – Pick up one of the reactants Type A such as $NaCl(aq)$ and add 20 drops to a set of four test tubes. Each tube should have now 20 drops of a reactant Type A.

☐ *Step 7:* – Pick up one of the reactants Type B such as $Ba(NO_3)_2(aq)$. Add 20 drops to the first test tubes containing the reactant A. Write down your observations on the Results table. Indicate if soluble as (S) or if insoluble as (I).

☐ *Step 8:* – Now pick another Type B reactant and add 20 drops to the second test tube containing the reactant A. Write down the result on the Results table as soluble (S) or insoluble (I). Do this until you run all the reactants Type B.

☐ *Step 9:* – Repeat this process for another Type A reactant, such as $NaBr(aq)$. Add 20 drops to a set of four test tubes and try adding different reactants Type B to each of these test tubes. Write down the result on the Results table as soluble (S) or insoluble (I). Once done, continue until you run all the reactants Type A.

Part B: Precipitate dissolution

☐ *Step 1:* – In a new, clean test tube mix 20 drops of $Ni(NO_3)_2$ and 20 drops of NaOH. Centrifuge the test tube for 3 seconds.

☐ *Step 2:* – Remove all liquid from the test tube with a dropper.

☐ *Step 3:* – Then add 20 drops of HCl to the precipitate and described the observations along the whole experiment indicating if the acid helps dissolve the precipitate.

> ⚠ CAUTION!
>
> ⚠ When using the centrifuge, do not attempt to stop the rotor with your fingers or anything else.
>
> ⚠ Sodium hydroxide, hydrochloric acid, acetic acid and ammonia can cause chemical burns.
>
> ⚠ Barium is poisonous. Wash your hands thoroughly after using it.

Part C: Acidic and Basic character

☐ *Step 1:* – Pick up some litmus paper, either red or blue; this is the dry paper. Indicate the color of the dry paper on the results.

☐ *Step 2:* – Have handy each of the following reactants: HCl, NH_3, $NH_4NO_3\,(aq)$, $CH_3COOH\,(aq)$ and $Na_2CO_3\,(aq)$.

☐ *Step 3:* – Add a drop of each reactant to a small piece of litmus. This is the wet paper. Write down the resulting color of the wet paper.

Part D: Acids-Base reactions

☐ *Step 1:* – Pick up some litmus paper, either red or blue. Indicate the color of the dry paper on the results. This is the dry paper.

☐ *Step 2:* – Have handy the set of reactants Type C: $NaOH\,(aq)$, $Na_2CO_3\,(aq)$ and $NH_3\,(aq)$

☐ *Step 3:* – Have handy the set of reactants Type D: $NH_4NO_3\,(aq)$, $HCl\,(aq)$, and $CH_3COOH\,(aq)$

☐ *Step 4:* – Pick up one of the reactants Type C such as $NaOH\,(aq)$ and add 20 drops to a set of four test tubes. Each tube will have now 20 drops of a reactant Type C.

☐ *Step 5:* – Pick up one of the reactants Type D such as $NH_4NO_3\,(aq)$. Add 20 drops to the first test tube containing the reactants C. Using litmus paper, test the acidity of the resulting solution. Write down the result on the Results table as acidic (A) or basic (B).

☐ *Step 6:* – Now pick another Type D reactant and add 20 drops to the second test tube containing the reactant C. Using litmus paper, test the acidity of the resulting solution. Write down the result on the Results table as acidic (A) or basic (B).

☐ *Step 7:* – Now, pick up another Type C reactant and follow the cycle above. Add 20 drops to a set of three test tubes. And try adding reactants Type D to each test tube. Using litmus paper, test the acidity of the resulting solution. Write down the acid/base character on the Results table as acidic (A) or basic (B). Do this until you run all the reactants Type D.

Pre-lab Questions

Ionic Reactions in Aqueous Solutions: Precipitation and acid-base reactions

1. When mixing sodium phosphate (Na_3PO_4) and lead(II) nitrate ($Pb(NO_3)_2$):

   (a) Predict the precipitate likely to be formed, if any.

   (b) Write the net ionic equation for the reaction.

2. Predict the Soluble character (*soluble* or *insoluble*) of the following substances.

Compound	Soluble character
$NaNO_3$	
NH_4Br	
$BaBr_2$	
$Ni(OH)_2$	
$BaCO_3$	

3. Write the balanced overall, complete ionic, and net ionic equations corresponding to each of the following reactions:

   (a) $BaCl_2(aq) + K_3PO_4(aq) \longrightarrow Ba_3(PO_4)_2(s) + KCl(aq)$

(b) $2\,HNO_3\,(aq) + Ba(OH)_2\,(aq) \longrightarrow Ba(NO_3)_2\,(aq) + 2\,H_2O\,(l)$
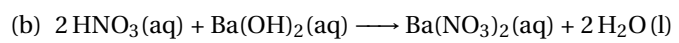
Results
EXPERIMENT

Ionic Reactions in Aqueous Solutions: Precipitation and acid-base reactions

Part A: Precipitation reactions

	NaCl	KCl	Na_2SO_4	NaBr
$Ni(NO_3)_2$				
$Pb(NO_3)_2$				
$AgNO_3$				
$Ba(NO_3)_2$				

(write *S* for soluble product and *I* for insoluble product. If insoluble, indicate the color of the precipitate)

Part B: Precipitate dissolution

Observations:

_____
_____
_____
_____
_____

Part C: Acids and bases

	Dry Litmus Color	Wet Litmus Color	Acid/Base
HCl			
NH$_3$			
NH$_4$NO$_3$(aq)			
CH$_3$COOH(aq)			
Na$_2$CO$_3$(aq)			
NaOH(aq)			

(write the color of the Litmus paper (red/blue).)

Part D: Acid-base reactions

	Dry Litmus Color	Wet Litmus Color		
		NaOH(aq)	Na$_2$CO$_3$(aq)	NH$_3$(aq)
NH$_4$NO$_3$(aq)				
HCl(aq)				
CH$_3$COOH(aq)				

(write the color of the Litmus paper (red/blue).)

Post-lab Questions

Ionic Reactions in Aqueous Solutions: Precipitation and acid-base reactions

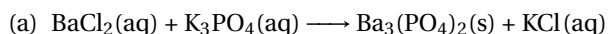1. When mixing barium nitrate $(Ba(NO_3)_2)$ and sodium hydroxide (NaOH):

    (a) Predict the precipitate likely to be formed, if any.

    (b) Write the net ionic equation for the reaction.

2. Complete the overall equation, and write the complete ionic equation and the net ionic equation for the following acid-base reaction. If the substance is a weak acid or base, leave it in its molecular form in the equations.

$$HF\,(aq) \quad + \quad NaOH\,(aq) \quad \longrightarrow$$
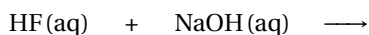
3. Identify the following compounds as a strong acid, weak acid, strong base, or weak base:

HF	_____	KOH	_____
HCl	_____	NH_3	_____
CH_3COOH	_____	NaOH	_____

EXPERIMENT

Acetic acid in vinegar: Titration of a weak acid

Goal

Weak acids are acids that do not dissociate completely, releasing only some of their hydrogen atoms into the solution. Acetic acid (CH_3COOH) is a very important weak acid, produced from the fermentation ethanol from the wine. Commercial acetic acid–what you know as vinegar–is just an aqueous solution of acetic acid. The goal of this experiment is to calculate the *molar concentration* of a sample of acetic acid utilizing a standard chemical procedure known as *titration*. To do that you will react the weak acid with a basic solution of sodium hydroxide (NaOH), which has a known concentration. You will also use phenolphthalein as *indicator*.

Materials

☐ a 10 mL transfer pipet and a 50 mL buret   ☐ a fisher clamp

☐ solutions of acetic acid and NaOH

☐ phenolphthalein   ☐ 4 125mL erlenmeyer

Background

A titration is a technique where a solution of known concentration–often times a base–is used to determine the unknown concentration of another solution–often times an acid [1]. Both substances react with each other in an acid-base reaction. The solution of known concentration is delivered carefully from a buret until an indicator–a third substance added to indicate the end of the titration–changes color. This experiment is a classic practice, previously implemented in numerous college chemistry laboratory manuals [2–5].

Concentration

We can express the composition of a solution as the *mass percentage* (%mass) of each component: solute and solvent.

$$\%mass = \frac{\text{mass of solute}}{\text{mass of solute + mass of solvent}} \times 100.\%$$

For example, if we dissolve 15 g of NaCl in 60. g of water, the total mass of the solution –solute plus solvent– is 75 g and the mass percentage of NaCl in the solution is $(15\,g/75\,g) \cdot 100\% = 20.\%$ NaCl. In chemistry, the molar concentration, c, of a solute in a solution, the *molarity* of the solute, is the moles of solute n present in a given volume, V, of the solution in liters. The units of molarity are moles per liter ($mol \cdot L^{-1}$), and it is denoted as M.

$$M = \frac{\text{moles of solute}}{\text{L of solution}} = \frac{n}{V}$$

Density, although not a measurement of concentration, is a property of a liquid that we can use to convert mass into volume or volume into mass. The formula for density d is:

$$d = \frac{\text{grams of solution}}{\text{mL of solution}}$$

Density can be used in combination with the molar mass, M_w, which converts mass into moles, to link the two expressions of concentration learned above, mass percent and Molarity.

$$\frac{c \times M_W}{d} \times 10^{-1} = \% \text{ mass}$$

use dimensional analysis to verify the expression above.

$$\frac{\frac{mol}{L} \times \frac{g}{mol}}{\frac{g}{mL}} \times \frac{1\,L}{1000\,mL} \times 100\% = 10^{-1}\%$$

Example

What is the molarity of a sodium hydroxide solution (NaOH, $M_W = 39.997\,g \cdot mol^{-1}$) prepared by dissolving 15.00 g of the solute in enough water to make 350.0 mL of solution?

- -

Answer: the molecular mass of NaOH is $39.997\,g \cdot mol^{-1}$ and the number of NaOH moles are:

$$n_{solute} = 15.00\,g \times \frac{1\,mol}{39.997\,g} = 0.3750\,mol$$

Do not forget to convert the volume from mL to L.

$$350.0\,mL \times \frac{1\,L}{1000\,mL} = 0.3500\,L$$

The molarity will be:

$$M = \frac{0.3750\,mol}{0.3500\,L} = 1.071\,M$$

Volumetric analysis

The determination of concentration by measuring volumes is called *volumetric analysis*. Titrations are volumetric analyses where a buret is used to add and measure the volume of one of the reactants. Acid-base titrations are extensively used chemical techniques employed to determine the solute concentration in a solution. In a *acid-base titration,* an acid reacts with a base by gradually adding one solution to the other. The volume of the second solution is known, and the volume of the first solution required for the complete reaction is measured. The formula to use in a titration is:

$$n_a \cdot c_a \cdot V_a = n_b \cdot c_b \cdot V_b,$$

where n_a and n_b is the number of protons and hydroxides, respectively, of the acid and the base involved in the titration. c_a and V_a are the concentration of the acid and the volume of acid employed, and c_b and V_b are the concentration of the base and the volume of base employed.

Example

A 25 mL solution of sulfuric acid, H_2SO_4 –which has two acidic protons– is titrated with NaOH 0.10 M. The end point for the reaction is reached after 40. mL of the NaOH solution are added. Find the molarity of the sulfuric acid solution.

- -

Answer: the balanced equation for the acid-base reaction is:

$$H_2SO_4\,(aq) \quad + \quad 2\,NaOH\,(aq) \quad \longrightarrow \quad 2\,H_2O \quad + \quad Na_2SO_4$$
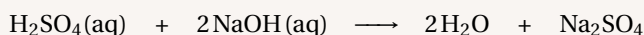
The acid (H_2SO_4) has two protons, hence $n_a = 2$. Its molarity, c_a, is unknown whereas the volume used, $V_a = 25\,mL$, is given.

The base (NaOH) has one hydroxide, hence $n_b = 1$. The base concentration, $c_b = 0.10\,M$, and volume, $V_b = 40.\,mL$, are given:

$$c_a = \frac{n_b \cdot c_b \cdot V_b}{n_a \cdot V_a}$$

$$c_a = \frac{1 \cdot 0.10\,M \cdot 40.\,mL}{2 \cdot 25\,mL} = 0.080\,M$$

An *indicator* is used to indicate the exact end of the reaction. The indicator chosen will have one color before the reaction is complete and a different color when the acid-base reaction finishes. For example in the reaction between acetic acid (CH_3COOH) and sodium hydroxide (NaOH):

$$CH_3COOH \quad + \quad NaOH \quad \longrightarrow \quad CH_3COONa \quad + \quad H_2O$$

using phenolphthalein as the indicator, the solution will be colorless before the competition of this reaction but pink after completion. At a specific point during the titration, a single drop of the NaOH from the buret will cause the solution being titrated to turn from colorless to a barely discernible pink color. This point is called the *endpoint*.

References

(1)   Crouch, D. S.D.W.F.L.H. S., *Fundamentals of analytical chemistry*; Nelson Education: 2013.

(2)   Beran, J. A., *Laboratory manual for principles of general chemistry*; John Wiley and Sons: 2010.

(3)   Ebbing, R. W.D. D., *Experiments in General Chemistry*; Houghton Mifflin Harcourt Publishing Company: 2004.

(4)   Russo, T., *Merrill Chemistry Lab Manual*; McGraw-Hill: 1998.

(5)   Holman, G. H. J., *Chemistry in Context*; McGraw-Hill Education: 2014.

Procedure

Getting started

☐ *Step 1:* – Obtain a 10 mL transfer pipet and a 50 mL buret with a stand and buret clamp.

☐ *Step 2:* – Obtain about 30 mL of acetic acid solution in a 50 mL beaker and about 80 mL of the NaOH solution in a clean, dry Erlenmeyer flask. Keep the NaOH solution containing Erlenmeyer closed with a rubber stopper.

☐ *Step 3:* – Clean your buret and fill it with the NaOH solution using a plastic funnel.

☐ *Step 4:* – Record the initial volume in the buret as zero. Mind you read the buret from the top and as you filled it up with NaOH the initial reading should be zero.

Good Lab Practice

✎ Learn how to execute an 🌍 *acid-base titration*.   ✎

Doing the titrations

☐ *Step 1:* – Pipet 5.00 mL of acetic acid into a clean 125 mL Erlenmeyer flask that has 20 mL of distilled water and 2 drops of phenolphthalein. Mind that you are placing the acid in the Erlenmeyer flask.

☐ *Step 2:* – Record the molarity of the NaOH solution indicated in the stock solution bottle in the results section.

☐ *Step 3:* – Fill up the buret with NaOH, the base.

☐ *Step 4:* – Place the flask under the buret. Use a piece of white paper under the flask to distinguish better the color change.

☐ *Step 5:* – Add the NaOH solution from the buret in 1 mL portions, while swirling the solution in the flask.

☐ *Step 6:* – The titration is completed when an addition of 1 mL causes the color to change from colorless to any shade of pink.

☐ *Step 7:* – Record the final buret volume.

☐ *Step 8:* – Repeat the steps above four times and average the resulting acetic acid concentration.

> ⚠ **CAUTION!**
>
> ⚠ All acids and bases in this experiment could cause chemical burns. ❗

Calculations

⓪ Record the molarity of the base, NaOH.

① Record the initial volume of the buret. This value is not necessarily 0.00 mL.

② Record the final volume of the buret, after you reached the end point.

③ The volumen of NaOH used should be: ②–①

④ You can calculate the molarity of the acetic acid solution by means of:

$$c_a = \frac{③ \cdot c_b}{5 \text{ mL}}$$

If the professor ask you to use a different volume of acetic acid, the value '5 mL' will need to be adjusted. where c_b is the given molarity of the NaOH solution.

⑤ Is the average of the 4 concentrations calculated.

$$\frac{\Sigma ④}{4}$$

STUDENT INFO

Name: Date:

Pre-lab Questions

Acetic acid in vinegar: Titration of a weak acid

1. A 10.00 mL sample of aqueous HCl requires 31.00 mL of 0.0900 M NaOH to reach the endpoint. What is the molar concentration of HCl? The equation for the reaction is:

$$HCl \quad + \quad NaOH \quad \longrightarrow \quad NaCl \quad + \quad H_2O$$
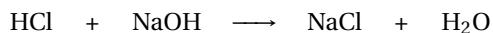
2. The molarity of a vinegar solution is 0.90 M. Calculate the number of acetic acid moles in 10. mL of this solution.

3. Phosphoric acid (H_3PO_4) is an acid with three protons. Suppose you titrate 5.00 mL of this acid with NaOH 0.10 M. Knowing that the end point is reached after 25.00 mL of the base is added, find the molarity of the acid solution.

Results
EXPERIMENT

Acetic acid in vinegar: Titration of a weak acid

Titration

(0) Molarity of NaOH, c_b =

		1	2	3	4
(1)	Initial Buret Volume (mL)				
(2)	Final Buret Volume (mL)				
(3)	NaOH Volume used (mL)				
(4)	CH_3COOH Concentration (M)				

(5) Mean CH_3COOH Concentration (M) =

Post-lab Questions

Acetic acid in vinegar: Titration of a weak acid

1. You need to prepare a sample containing 0.20 g of $CuSO_4$ from a solution that is 10.% $CuSO_4$ by mass. What mass of solution do you need?

2. A 10.00 mL sample of aqueous H_2SO_4 requires 20.00 mL of 0.201 M NaOH to reach the endpoint. Calculate the molarity of H_2SO_4.

3. You titrate a vinegar sample–an acetic acid solution in water–with 0.30 M NaOH. Using 10. mL of vinegar, you reach the endpoint after 10. mL of the bases are added. Indicate the molarity of the acetic acid solution.

15 Identity of Insoluble Precipitate: Gravimetry

<div align="center">

EXPERIMENT

Identity of Insoluble Precipitate: Gravimetry

</div>

Goal

The goal of this experiment is to study the reaction between barium sulfate ($Ba(NO_3)_2$) and sulfamic acid (NH_2SO_3H) in hot water to produce barium sulfate ($BaSO_4$). This reaction is called hydrolysis. Using hot gravity filtration and through a gravimetric analysis, you will calculate the reaction yield of the reaction.

Materials

- ☐ $Ba(NO_3)_2$ and NH_2SO_3H
- ☐ A 250 mL beaker and a graduated cylinder
- ☐ Stirring rod and rubber policeman (see the Figure)

- ☐ Filter paper and funnel
- ☐ Stand and iron ring
- ☐ Tongs

Background

Sulfamic acid (NH_2SO_3H), also known as amino sulfonic acid, is a very common chemical used to remove grout and mortar haze, as well as rust and mineral deposits [1]. It can indeed be found in many home improvement retail stores [2]. The hydrolysis of this acid (the reaction with water at high temperature) produces ammonium hydrogen sulfate:
$NH_2SO_3H + H_2O \longrightarrow NH_4HSO_4$ The hydrolysis of sulfamic acid to give sulfates can be used to precipitate barium in the form of barium sulfate [3].

$$Ba(NO_3)_2 \quad + \quad NH_2SO_3H \quad + \quad H_2O \quad \longrightarrow \quad BaSO_4 \quad + \quad NH_4NO_3 \quad + \quad HNO_3$$
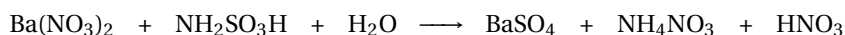
By weighing the amount of precipitate, you can trace the amount of barium in the solution. In this experiment, you will react sulfamic acid with sodium nitrate in hot water to produce a precipitate of barium sulfate, focusing on the yield of the reaction. The advantage of using sulfamic acid instead of other types of sulfur acids, such as sulfuric acid, is that sulfamic acid produces a coarse crystalline precipitate with fewer impurities [4].

Reaction yield

Stoichiometric calculations can predict the amount of product that will be formed in a chemical reaction. However, chemical reactions are not perfect, and oftentimes reactions do not proceed to be fully completed, and the amount of product predicted is not the one obtained in the experiment. The *yield* of a chemical reaction refers to the amount (mass or moles) of the product obtained in the experiment (the actual) with respect to the theoretical quantity obtained according to stoichiometric calculations (the theoretical).

$$\% \text{Yield} = \frac{\text{Actual amount}}{\text{Theoretical amount}} \times 100\%$$

Example

We mix 2.0 moles of $Ba(NO_3)_2$ with an excess of H_2SO_4 to produce a $BaSO_4$ precipitate. We obtain 1.0 moles of $BaSO_4$:

$$Ba(NO_3)_2(aq) \quad + \quad H_2SO_4(aq) \quad \longrightarrow \quad BaSO_4(s) \quad + \quad 2\,HNO_3(aq)$$

Calculate the reaction yield.

Answer: according to stoichiometric calculations, (coefficients in the balanced chemical equation) 2 moles of $Ba(NO_3)_2$ produce 2 moles of $BaSO_4$. Since we start with 2.0 moles of $Ba(NO_3)_2$, one would expect to also produce 2 moles of $BaSO_4$. But the actual number of moles of $BaSO_4$ is 1.0 mole. Hence the yield is

$$\% \, Yield = \frac{Actual\ amount}{Theoretical\ amount} \times 100\% = \frac{1.0\ moles}{2.0\ moles} \times 100\% = 50\%.$$

Limiting Reactant

In a chemical reaction involving two reactants normally one of the reactants limits the reactions, which means, limits the amount of product generated. This means that the chemical reaction will proceed as long as there is a limiting reactant. After all limiting reactant has reacted the process will stop and an excess of the other reactant will remain unused. Most of the time is not so obvious to identify the limiting reactant and one needs to do some basic calculations to identify the limiting reactant as well as the excess reactant.

Example

We mix 2 moles of NaOH with an 1.5 moles of $MgCl_2$:

$$2\,NaOH(aq) \quad + \quad MgCl_2(aq) \quad \longrightarrow \quad Mg(OH)_2(s) \quad + \quad 2\,NaCl(aq)$$
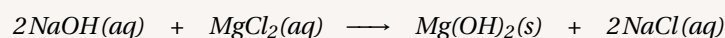
Identify the limiting and the excess reactant.

Answer: we first choose one of the reactants, for example NaOH, to then convert the number of moles of that reactant into how many moles will be needed from the other reactant, according to the stoichiometry of the balanced chemical equation:

$$n_{MgCl_2} = 2 \text{ moles of NaOH} \times \frac{1 \text{ mole } MgCl_2}{2 \text{ moles NaOH}} = 1 \text{ mole of } MgCl_2$$

This means that to react with 2 moles of NaOH we would need 1 mole of $MgCl_2$. Because we have indeed 1.5 moles of $MgCl_2$ that means we do have enough $MgCl_2$ and hence NaOH should be the limiting reactant. $MgCl_2$ would be the excess reactant.

Filtration

Filtration is a technique employed in chemistry to separate an insoluble solid compound (the precipitate) from the liquid. The mixture containing the solid is poured through a fine mesh (the filter) and gravity makes the liquid go through the filter, while the solid remains on it. When the mixture containing the solid is very hot, we call the filtration procedure: "hot gravity filtration". Different types of filter paper are available with a variety of porosities to separate different types of solids. Filter paper must be folded before proceeding with the filtration, forming a cone. To do this, one needs to first fold the filter in half, and then in half again, as shown in the Figure. Do not press the tip of the cone while folding, because it will weaken the paper.

Gravimetric Analysis

Gravimetric analysis is a technique used to find out how much of a metal ion, such as barium, is in a solution. Think about a water sample containing Ba^{2+} dissolved. To determine how much barium is in the solution it would be necessary to separate it from the solvent. One way to separate the metal would be to precipitate the Ba^{2+} ions through a chemical reaction. Later, by filtering off and weighting the precipitate (containing all the barium) you would be able to determine the amount of Ba^{2+} in the original solution. A successful precipitation reaction would need to proceed to completion to precipitate the complete amount of Ba^{2+} ions in the solution.

References

(1) pubchem., `https://pubchem.ncbi.nlm.nih.gov`, Accessed: 2017-04-22.

(2) www.homedepot.com/., `http://www.homedepot.com/`, Accessed: 2017-04-22.

(3) Wagner, W. F.; Wuellner, J. A. *Analytical Chemistry* **1952**, *6*, 1031–1032.

(4) Notley, J. M. *Journal of Applied Chemistry and Biotechnology* **1973**, *10*, 717–723.

Procedure

Initiating the Reaction

☐ *Step 1:* – You will need 1.2 g to 1.5 g of $Ba(NO_3)_2$ and 2.3 g to 2.5 g NH_2SO_3H. Use separate weighing boats to get approximate amounts of the 2 reactants.

☐ *Step 2:* – Weight a clean, dry, 250 mL beaker and record the exact mass in the Results section.

☐ *Step 3:* – Transfer the $Ba(NO_3)_2$ samples to a 250 mL beaker and record the mass of the beaker with the compound in the Results section.

☐ *Step 4:* – Transfer the NH_2SO_3H samples to the beaker and record the mass of the beaker with the two compounds in the Results section.

☐ *Step 5:* – Add 150 mL of distilled water and mix with a glass stirring rod.

☐ *Step 6:* – Prepare a stand with two metal rings and a wire gauze. The second metal ring will serve as a fence to prevent the beaker from falling. Get a Bunsen Burner ready. Set the height of the metal rings according to the size of the flame. It is important to know that the size of the flame can be reduced using the burner valve.

☐ *Step 7:* – If your instructor wants you to use a hot place you will only need a single metal ring. Mind the beaker might jump if you use too much heat. If this happens reduce the heat immediately.

☐ *Step 8:* – On a different spot of your lab table, prepare a smaller metal ring with a funnel for the filtration.

☐ *Step 9:* – Place the solution on the wire gauze. Leave the stirring rod inside. Bring the water to a boil and reduce the size of the flame when it starts boiling to avoid spills. Allow the solution to boil for about 30 min. Make sure the amount of liquid ($\approx$ 150 mL) does not change, by adding extra water with the wash bottle. Stir the solution occasionally.

> ⚠ **CAUTION!**
>
> ⚠ Barium solutions are toxic. Make sure you to wash your hands thoroughly after working with Barium compounds.
>
> ⚠ If your beaker starts jumping when using a hot place, immediately reduce the heat.

Filtering and weighting of the dry precipitate

☐ *Step 10:* – After 30 minutes of boiling, turn off the Bunsen Burner and cool the beaker to room temperature. Do not move the beaker until you can handle it with your hand without burning it.

☐ *Step 11:* – Obtain a piece of filter paper and record its mass.

☐ *Step 12:* – When the reaction mixture containing the precipitate is cooled, proceed to filter the mixture. Make sure no trace of precipitate remains in the beaker. Use a wash bottle to rinse any product left.

☐ *Step 13:* – Transfer the filter paper and its contents to a watch glass labeled with your name on it. Make sure the precipitate does not touch the glass.

☐ *Step 14:* – Place the beaker in an oven at 90°C for at least 1 hour, until the paper is fully dry. Make sure you use gloves to protect yourself from the heat.

☐ *Step 15:* – Remove the dry filter paper with dry precipitate from the oven after 1 hour, always using gloves. Make sure the filter paper is completely dry. Leave the watch glass in the oven, so that you can place the paper with the precipitate directly in the tare balance using a cool watch glass. Record the mass of the filter paper and its content.

☐ *Step 16:* – Calculate the % Yield.

Calculations

① Measured using the balance.

② Measured using the balance.

③ Measured using the balance.

④ Calculate mass by subtraction: ② – ①

⑤ Calculate mass by subtraction: ③ – ②

⑥ Measured using the balance.

⑦ Measured using the balance.

⑧ Calculate mass by subtraction: ⑦ – ⑥

⑨ Convert ④ using the reciprocal of the molar mass of the corresponding chemical compound.

⑩ Convert ⑤ using the reciprocal of the molar mass of the corresponding chemical compound.

⑪ Using the balanced chemical equation and the number of moles calculated in ⑨ and ⑩ , calculate the limiting reactant as in the example and in the pre-lab.

⑫ Convert moles to moles, and to mass: ⑨ × 261.337

⑬ The yield is given by
$$\%Yield = ⑧ / ⑫ \times 100\%$$

Pre-lab Questions

Identity of Insoluble Precipitate: Gravimetry

1. Explain what to do if you warm up a beaker filled with liquid using a hot plate and it starts to jump.

2. For the hydrolysis of sulfamic acid NH_2SO_3H (MW 97.1 $g \cdot mol^{-1}$) reacting with $Ba(NO_3)$ (MW 261.34 $g \cdot mol^{-1}$) to produce $BaSO_4$ (MW 233.38 $g \cdot mol^{-1}$), we use a solution containing 1.50 g $Ba(NO_3)_2$ and 2.50 g of NH_2SO_3H :

$$Ba(NO_3)_2(aq) \; + \; NH_2SO_3H(aq) \; + \; H_2O(l) \; \longrightarrow \; BaSO_4(s) \; + \; NH_4NO_3(aq) \; + \; HNO_3(aq)$$
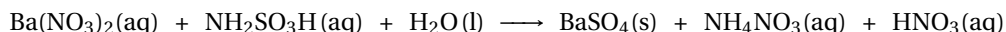
   (a) Use the expression below and calculate the grams of sulfamic acid needed to react with 1.50 g of $Ba(NO_3)_2$:

$$\text{grams } NH_2SO_3H = 1.50 \text{grams of } BaNO_3 \; \times \frac{\rule{2cm}{0.4pt}\text{mol } BaNO_3}{\rule{2cm}{0.4pt}\text{g } BaNO_3} \times \frac{\rule{2cm}{0.4pt}\text{mol } NH_2SO_3H}{\rule{2cm}{0.4pt}\text{mol } BaNO_3}$$

$$\times \frac{\rule{2cm}{0.4pt}\text{g } NH_2SO_3H}{\rule{2cm}{0.4pt}\text{mol } NH_2SO_3H} = \rule{2cm}{0.4pt}\text{g } NH_2SO_3H \text{ needed to react}$$

   (b) Identify how many grams of sulfamic acid you have and see if you have more or less sulfamic acid than the amount you need. If you have more than you need, then $BaNO_3$ is the limiting reactant. If you do not have enough, then sulfamic acid should be the limiting reactant.

   (c) Assuming $BaNO_3$ is the limiting reactant, calculate the grams of $BaSO_4$ produced using the following expression:

$$1.5 \text{ grams of } BaNO_3 \; \times \frac{\rule{2cm}{0.4pt}\text{mol } BaNO_3}{\rule{2cm}{0.4pt}\text{g } BaNO_3} \times \frac{\rule{2cm}{0.4pt}\text{mol } BaSO_4}{\rule{2cm}{0.4pt}\text{mol } BaNO_3} \times \frac{\rule{2cm}{0.4pt}\text{g } BaSO_4}{\rule{2cm}{0.4pt}\text{mol } BaSO_4} =$$

$$= \rule{2cm}{0.4pt}\text{g } BaSO_4 \text{ produced}$$

   (d) Knowing that the chemical reaction produces 1.0 g of $BaSO_4$. Calculate the % yield of the reaction.

Results
EXPERIMENT

Identity of Insoluble Precipitate: Gravimetry

1	Mass of empty, clean, dry 250 mL beaker (g)	
2	Mass of beaker + $Ba(NO_3)_2$ (g)	
3	Mass of beaker + $Ba(NO_3)_2$ + NH_2SO_3H (g)	
4	Mass of $Ba(NO_3)_2$ (g)	
5	Mass of NH_2SO_3H (g)	
6	Mass of dry, unused filter paper (g)	
7	Mass of filter paper + precipitate after oven (g)	
8	Mass of dry precipitate (g)	
9	Moles of $Ba(NO_3)_2$ (g)	
10	Moles of NH_2SO_3H (g)	
11	Limiting reactant	
12	Theoretical mass of precipitate	
13	% Yield=$\frac{\text{Actual mass of precipitate}}{\text{Theoretical mass of precipitate}} \times 100\%$	

STUDENT INFO

Name: Date:

Identity of Insoluble Precipitate: Gravimetry

1. For the reaction of NaOH (M_W 39.997 $g \cdot mol^{-1}$) with $MgCl_2$ (M_W 95.221 $g \cdot mol^{-1}$) to produce $Mg(OH)_2$ (M_W 53.320 $g \cdot mol^{-1}$), we use a solution containing 1.00 g NaOH and 2.00 g of $MgCl_2$:

$$2\,NaOH\,(aq) \quad + \quad MgCl_2\,(aq) \quad \longrightarrow \quad Mg(OH)_2\,(s) \quad + \quad 2\,NaCl\,(aq)$$
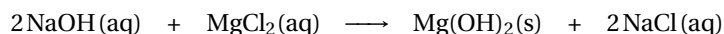
(a) Use the expression below and calculate the grams of $MgCl_2$ needed to react with 1.00 g of NaOH :

$$\text{grams } MgCl_2 = 1.00 \text{ grams of NaOH } \times \frac{\rule{1cm}{0.4pt}\ \text{mol NaOH}}{\rule{1cm}{0.4pt}\ \text{g NaOH}} \times \frac{\rule{1cm}{0.4pt}\ \text{mol } MgCl_2}{\rule{1cm}{0.4pt}\ \text{mol NaOH}}$$

$$\times \frac{\rule{1cm}{0.4pt}\ \text{g } MgCl_2}{\rule{1cm}{0.4pt}\ \text{mol } MgCl_2} = \rule{1cm}{0.4pt}\ \text{g } MgCl_2 \text{ needed to react}$$

(b) Identify how many grams of $MgCl_2$ you have and see if you have more or less $MgCl_2$ than the amount you need. If you have enough, then NaOH is the limiting reactant. If you do not have enough, then $MgCl_2$ should be the limiting reactant.

(c) Assuming NaOH is the limiting reactant, calculate the grams of $Mg(OH)_2$ produced using the following expression:

$$1 \text{ grams of NaOH } \times \frac{\rule{1cm}{0.4pt}\ \text{mol NaOH}}{\rule{1cm}{0.4pt}\ \text{g NaOH}} \times \frac{\rule{1cm}{0.4pt}\ \text{mol } Mg(OH)_2}{\rule{1cm}{0.4pt}\ \text{mol NaOH}} \times \frac{\rule{1cm}{0.4pt}\ \text{g } Mg(OH)_2}{\rule{1cm}{0.4pt}\ \text{mol } Mg(OH)_2} =$$

$$= \rule{1cm}{0.4pt}\ \text{g } Mg(OH)_2 \text{ produced}$$

(d) Knowing that the chemical reaction produces 1.00 g of $Mg(OH)_2$. Calculate the % yield of the reaction.

EXPERIMENT

Thermochemistry and Hess's Law: Heat of neutralization

Goal

The goal of this experiment is to measure the heat involved in two acid-base chemical reactions: the neutralization of NaOH and NH_3 with HCl. You will determine the enthalpy change for the two acid-base neutralization reactions using a coffee-cup calorimeter and a thermometer. By measuring the heat released or consumed you will be able to calculate the enthalpy change ΔH for both reactions.

Materials

□ A coffee-cup calorimeter   □ HCl (aq), NH_3 (aq) and NaOH (aq) solutions

□ A thermometer and a lit for the calorimeter

Background

Reactants and products in a chemical reaction normally have a different energy, and this difference in energy is reflected by a release or absorption of heat. Utilizing a coffee-cup calorimeter–a constant pressure calorimeter–and a thermometer, we can measure temperature changes produced by a chemical reaction. When a reaction produces heat, the temperature inside the calorimeter will rise, whereas the temperature will decrease if the reaction consumes heat. These energy changes are related to the enthalpy change–the heat exchanges at constant pressure–of the chemical reaction. This is a classical college chemistry experiment implemented in many lab manuals with different choices of acids and bases [1–4].

Measurement of heat

Heat–thermal energy q–is the energy transferred as a result of a temperature difference. q flows from regions of high-temperature to low-temperature regions. Heat is connected to change in temperature ΔT through the specific heat C:

$$q = m \cdot C \cdot \Delta T$$

where m is the mass of the object. Large heat capacity means that a small supply of heat produces a big temperature increase. Metals for example have small specific heats. For aqueous solutions, C is the specific heat of water, $4.184 \, J \cdot ° \, C^{-1} \cdot g^{-1}$, and assuming the density of the solution is $1.00 \, g \cdot mL^{-1}$ the energy change due to a change in temperature $\Delta T = T_f - T_i$ is given by:

$$q = \left(d \cdot V \right) \cdot C \cdot \Delta T$$

where V is the volume of solution in mL and the temperatures are in $°C$.

Enthalpy change of a chemical reaction

The energy change in a chemical reaction measured at constant pressure–in an open container–is called enthalpy change, ΔH. All chemical reactions are accompanied by a change in energy. For example, the reaction between an acid and a base is called neutralization, and for the case of HCl and NaOH is the following reaction:

$$\text{HCl} + \text{NaOH} \longrightarrow \text{NaCl} + \text{H}_2\text{O} \qquad \Delta H_1 = -52 \; KJ$$
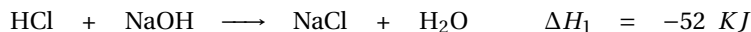
The heat involved in this process is -52 kJ. The negative sign means that the heat is being released. Reactions that release heat are called exothermic ($\Delta H < 0$), whereas reactions that absorb heat are called endothermic ($\Delta H > 0$). The heat released in a chemical reaction depends on the external conditions. The standard reaction enthalpy is the reaction enthalpy when reactants in their standard states change into products in their standard states, that is its pure form at exactly 1 bar. The enthalpy of an element in its standard state is always zero. For example for $H_2(g)$ $\Delta H = 0$.

Using a coffee-cup calorimeter

Two nested polystyrene cups–coffee cups–make a good constant–pressure calorimeter, as polystyrene is a good heat insulator. With a coffee-cup calorimeter and a thermometer, we can measure the enthalpy change ΔH of a chemical reaction happening inside the calorimeter: the heat q consumed or released inside a calorimeter is related to the enthalpy change of the reaction by:

$$\Delta H_r + q = 0$$

Often heat flows through the walls of calorimeters, as these devices are never perfect. To take into account this loss of heat, we can use the following formula:

$$\Delta H_r + q + C_{Cal}\Delta T = 0$$

where C_{Cal} is the calorimeter constant and it is calculated to be $C_{Cal} = 10.0 \, J \cdot^\circ C^{-1}$ for a standard calorimeter. It accounts for the energy absorbed by the calorimeter. Because of this effect, we will not be able to observe the highest (or lowest) temperature that could have been achieved in a perfectly insulated calorimeter, as when the temperature in a calorimeter starts rising due to a reaction, the heat starts leaving the cup. We can compensate for this effect and estimate the temperature when the heat leakage is minimal, by plotting temperature as a function of time and then extrapolating the values to the beginning of the reaction.

Example

When 10.00 ml of nitric acid 1.0 M, HNO_3, reacts with excess of NaOH:
$HNO_3(aq) + NaOH(aq) \longrightarrow NaNO_3(aq) + H_2O(aq)$
in a coffee-cup calorimeter with a heat capacity of 10. $J\cdot^\circ C^{-1}$, the temperature of the calorimeter rises by $10.0^\circ C$. Calculate the reaction enthalpy for this process assuming the density of the mixture is $1.00\frac{g}{mL}$.

- -

Answer: the heat absorbed by the calorimeter is the heat exchanged during the chemical reaction:

$$\Delta H_r + \left(d \cdot V\right) \cdot C \cdot \Delta T + C_{Cal} \cdot \Delta T = 0$$

where V is the volume used of nitric acid. Plugging all the values:

$$\Delta H_r + \left(1.00 \, g \cdot mL^{-1} \cdot 10.00 \, mL\right) \cdot 4.184 \, J \cdot^\circ C^{-1} \cdot g^{-1} \cdot 10.0^\circ C^{-1} + 10. \, J \cdot^\circ C^{-1} \cdot 10.0^\circ C = 0$$

Solving for ΔH_r we have $-520 \, J$. The number of moles of HNO_3 is:

$$n = 10.00 \, mL \times \frac{1.0 \, mol}{L} = 0.010 \, mol$$

hence $\Delta H_r = -5,200 \, J/mol = -52 \, kJ/mol$

Hess's Law

Hess's law states that the total enthalpy change during a chemical reaction is the same whether the reaction results in one or several steps. For example:

$$HCl \ + \ NaOH \ \longrightarrow \ NaCl + H_2O \qquad\qquad \Delta H_1$$

$$NH_3 \ + \ HCl \ \longrightarrow \ NH_4Cl \qquad\qquad \Delta H_2$$

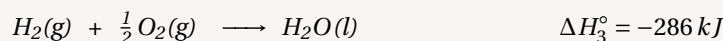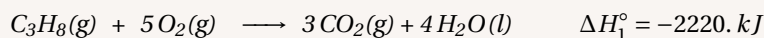$$NaOH \ + \ NH_4Cl \ \longrightarrow \ NaCl + NH_3 + H_2O \qquad\qquad \Delta H_3$$
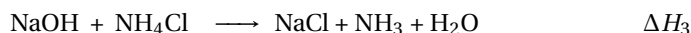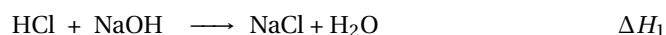
You might have noted there is a relationship among these equations: if you reverse the second equation and add it to the first equation, the third equation is generated. This provided the basis for Hess's law and you will find you can predict the enthalpy change for the third reaction by combining the enthalpy change for the first and second reactions:

$$\Delta H_3 = \Delta H_1 - \Delta H_2$$

Example

Calculate the standard enthalpy of the reaction $3\,C\,(gr) + 4\,H_2\,(g) \longrightarrow C_3H_8\,(g)$, ΔH_R°, *using the following data:*

$$C_3H_8\,(g) \ + \ 5\,O_2\,(g) \ \longrightarrow \ 3\,CO_2\,(g) + 4\,H_2O\,(l) \qquad \Delta H_1^{\circ} = -2220.\,kJ$$

$$C\,(g) \ + \ O_2\,(g) \ \longrightarrow \ CO_2\,(g) \qquad \Delta H_2^{\circ} = -394\,kJ$$

$$H_2\,(g) \ + \ \tfrac{1}{2}\,O_2\,(g) \ \longrightarrow \ H_2O\,(l) \qquad \Delta H_3^{\circ} = -286\,kJ$$

- - - - - - - - - -

Answer: Using Hess's law, we can build up the reaction by reversing the first reaction, multiplying the second reaction times 3, multiplying the reaction 3 times 4, and adding them. If we do the same to the enthalpies we obtain:

$$\Delta H_R^{\circ} = 3 \cdot \Delta H_2^{\circ} + 4 \cdot \Delta H_3^{\circ} - \Delta H_1^{\circ} = -106\,kJ$$

---

How to find extrapolated temperature

A calorimeter is ideally an isolated system. That means the heat produced does not leave the cup. If a reaction is exothermic–if it produces heat–the temperature inside the calorimeter will increase. Differently, for an endothermic reaction–a reaction that consumes heat–the temperature inside the calorimeter will decrease. When the reaction stops, as there is no more heat being produced or consumed, the temperature inside the calorimeter will change. To estimate the temperature at that point, we need to extrapolate the temperature inside the calorimeter. If you plot temperature versus time you will see that for early times, the temperature inside a calorimeter in which a reaction happens changes abruptly. After that, the temperature slowly decreases with time. To calculate the temperature when the reaction stops you need to connect the dots of the later stages, when the reaction already has stopped. We call this interpolation. All these points will be part of a line. If you extend the line, it will cross the Y-axis, and that crossing point is the temperature at which the reaction has stopped. We call this the extrapolation method.

Example

The following data reports the temperature inside a cup calorimeter. Indicate whether the reaction is exothermic or endothermic and calculate the temperature when the reaction stops employing the extrapolation method.

t (min)	0	1	2	3	4	5	6	7	8	9	10
T (°C)	10	10	20	28	29	30	29.5	29	28.5	27.5	27

Answer: if we plot temperature versus time we will see the two different regions of the plot. In early times, the temperature increased and hence the reaction in the calorimeter was exothermic. At later times, the reaction stops and temperature decreases due to the lack of heat release. We will use the last points and connect them with a line. The crossing point with the Y axis is 33 °C and that is the final temperature of the reaction.
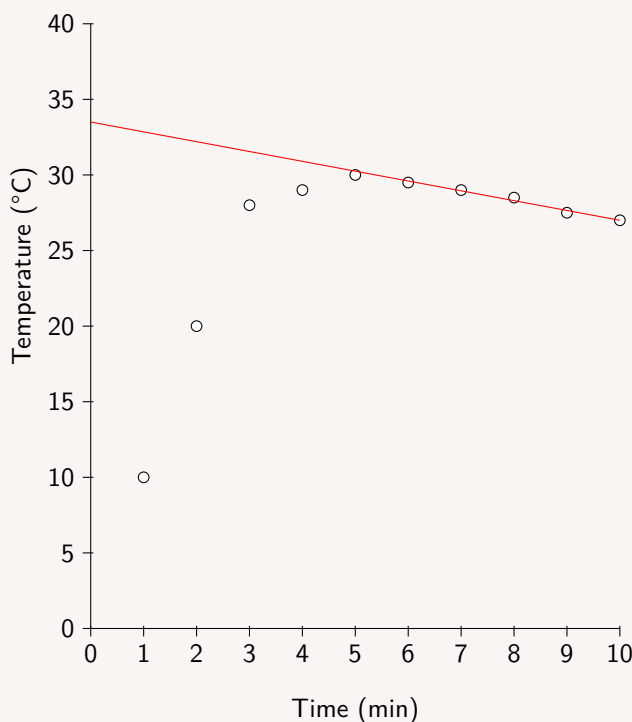
Procedure

Measuring ΔH for the HCl – NaOH pair

☐ *Step 1:* – Obtain a coffee-cup calorimeter with a thermometer and a plastic lid.

☐ *Step 2:* – Place 50.0 mL of HCl and 50.0 ml of NaOH in separate graduated cylinders. Make sure the cylinders are dry and clean.

☐ *Step 3:* – Write down the concentration of the acid and the base in the results section.

☐ *Step 4:* – Measure the temperature of each of these solution. If you use the same thermometer for both solutions, make sure you clean the thermometer in between measurements as the chemicals will react. Record the mean of the two temperatures as your initial temperature t_i.

☐ *Step 5:* – Get the timer ready. Time should be taken as soon as the second reactant is added.

☐ *Step 6:* – Add the acid to the calorimeter. If you are using a magnetic stirrer set the speed to a medium speed. Then add the base. Start the timer immediately and place the lid on the calorimeter.

☐ *Step 7:* – Record the temperature for 4 min. Do not stop the timer during the experiment. Record the results in the results table.

☐ *Step 8:* – Plot the temperature of the calorimeter against time and use a straight line to extrapolate your results against the starting time (time = 0 s). Record the extrapolated temperature as t_f.

☐ *Step 9:* – Calculate the change of enthalpy, ΔH.

> **Good Lab Practice**
>
> ✍ Make sure you always use the same thermometer throughout the experiment so that the results are consistent.

Measuring ΔH for the $HCl - NH_3$ pair

☐ *Step 10:* – Obtain a coffee-cup calorimeter with a thermometer and a plastic lid.

☐ *Step 11:* – Place 50.0 mL of HCl and 50.0 ml of NH_3 in separate graduated cylinders. Make sure the cylinders are dry and clean.

☐ *Step 12:* – Measure the temperature of each of these solution. If you use the same thermometer for both solutions, make sure you clean the thermometer in between measurements as the chemicals will react. Record the mean of the two temperatures as your initial temperature t_i.

☐ *Step 13:* – Get the timer ready. Time should be taken as soon as the second reactant is added.

☐ *Step 14:* – Add the acid, HCl to the calorimeter. If you are using a magnetic stirrer set the speed to a medium speed. Then add the base, NH_3. Start the timer immediately and place the lid on the calorimeter.

☐ *Step 15:* – Record the temperature for 4 min. Do not stop the timer during the experiment. Record the results in the results table.

☐ *Step 16:* – Plot the temperature of the calorimeter against time and use a straight line to extrapolate your results against the starting time (time = 0 s). Record the extrapolated temperature as t_f.

☐ *Step 17:* – Calculate the change of enthalpy, ΔH.

> **⚠CAUTION!**
>
> ⚠ All acids and bases in this experiment, HCl, NaOH and NH_3, can cause chemical burns.   !

Calculations

① This is the initial temperature before you mix the reagents.

② This is the final temperature after the reaction stops. You need to plot the data and extrapolate to get this value.

③ This is the number of moles reacting: $c_{acid} * 50/1000$

④ You can calculate the enthalpy change (ΔH) by means of the following formula:

$$\Delta H = -\frac{1}{③} \cdot \left(4.184 \cdot 1.0 \cdot 100.0 \cdot (② - ①) + 10 \cdot (② - ①)\right)$$

STUDENT INFO

Name: Date:

Pre-lab Questions

Thermochemistry and Hess's Law: Heat of neutralization

1. An acid and a base are mixed in a coffee-cup calorimeter. We recorded the temperatures with time and the data is given below. The initial temperature, t_i was 25.0 °C.

t (s)	30	60	90	120	150	180	210	240
T (°C)	40.2	40.6	40.5	40.4	40.3	40.2	40.1	40.0

(a) Plot T vs. t. Obtain t_f, the final temperature after the reaction starts, by extrapolating to the time of mixing (time = 0 s) with a straight line.
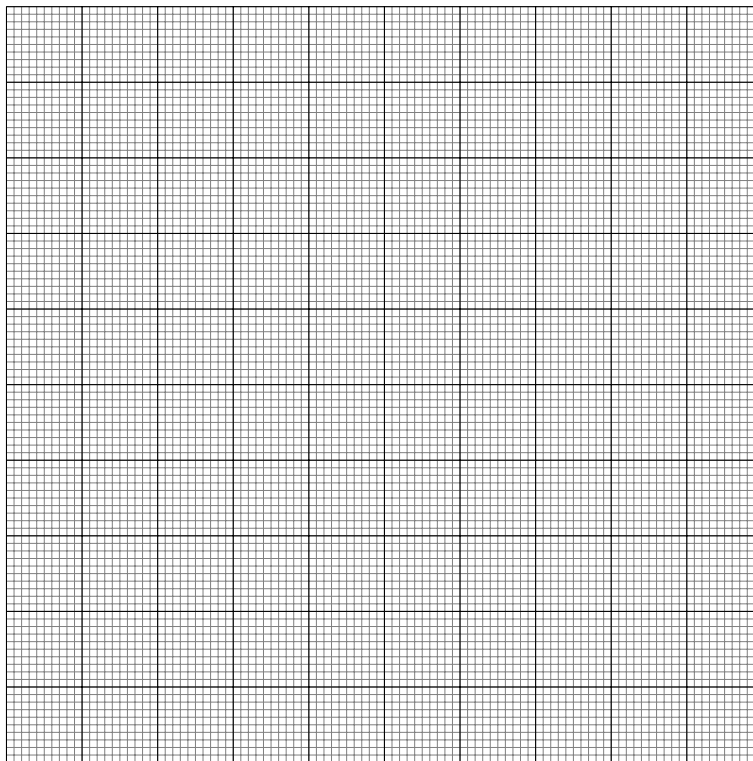
$$t_f \quad =$$

(b) Indicate whether this is an exothermic or an endothermic reaction.

(c) Explain the rise and fall of the temperature inside the calorimeter.

Thermochemistry and Hess's Law: Heat of neutralization

Results
EXPERIMENT

$c_{acid} =$ $c_{base} =$

HCl – NaOH Trial 1

t (s)	0	30	60	90	120	150	180	210	240
T (°C)									

T_i (°C)	T_f (°C)	n (moles)	$\Delta H (kJ/mol)$
①	②	③	④

HCl – NaOH Trial 2

t (s)	0	30	60	90	120	150	180	210	240
T (°C)									

T_i (°C)	T_f (°C)	n (moles)	$\Delta H (kJ/mol)$

Average $\Delta H (kJ/mol) =$

$c_{acid} =$ $c_{base} =$

HCl–NH₃ Trial 1

t (s)	0	30	60	90	120	150	180	210	240
T (°C)									

T_i (°C)	T_f (°C)	n (moles)	$\Delta H (kJ/mol)$
①	②	③	④

HCl–NH₃ Trial 2

t (s)	0	30	60	90	120	150	180	210	240
T (°C)									

T_i (°C)	T_f (°C)	n (moles)	$\Delta H (kJ/mol)$

Average $\Delta H (kJ/mol) =$

Figure 6: HCl – NaOH

Figure 7: HCl–NH$_3$

Thermochemistry and Hess's Law: Heat of neutralization

1. 5.0 ml of a 1 M acid reacts with 5.0 ml of 1 M base in a coffee-cup calorimeter with a heat capacity of 10. $J \cdot (°C)^{-1}$. Calculate the rise in temperature of the calorimeter assuming the density of the mixture is $1 \frac{g}{ml}$ and given that $\Delta H_r = 50. \, kJ/mol$.

2. When 5.0 mL of 1.00 M HNO_3 solution at 25.0 °C reacts with 5.0 mL of 1.00 M NaOH solution at the same temperature in a calorimeter, the temperature of the resulting mixture increases to 31.8 °C. Assuming that the calorimeter absorbs only a negligible quantity of heat, that the specific heat capacity of the solution is 4.18 $J \cdot (°C)^{-1} \cdot g^{-1}$, and that the density of the final solution is 1.0 $g \cdot mL^{-1}$. Calculate the enthalpy of neutralization.

3. Calculate the energy to increase the temperature of a 5 g piece of iron from 25.0 °C to 75.0 °C, knowing the specific heat capacity of iron is 0.45 $J \cdot (°C)^{-1} \cdot g^{-1}$.

EXPERIMENT

Molar Mass of a Volatile Liquid

Goal

The goal of this experiment is to calculate the molar mass (molecular weight, MW) of a volatile gas using the Dumas method, employing the formula of the ideal gases and by measuring the mass of the gas.

Materials

☐ an unknown liquid

☐ a 600 mL beaker

☐ a 125 mL Erlenmeyer

☐ a 10 and 100 mL graduated cylinders

☐ a hot plate

☐ a thermometer

☐ a stand and iron ring

☐ a rubber band and aluminum foil

Background

Gases have a very different behavior than liquids or solids. In a gas, particles (atoms, or molecules) are far apart from each other, while in solids or liquids, particles are closer to each other and interact through stronger forces. As a consequence, gases are less dense–have fewer particles per volume unit–than liquids or solids and are easy to compress as well. Think for example of how easy is to compress a balloon full of air, even with your own hands. In this process the gas particles, which are far away from each other, get closer together. Another characteristic exclusive of gases is that they will fill the volume of their container. All these properties of gases are explained by the kinetic molecular theory of gases. Gases or vapors can be easily produced by boiling a liquid. But even below their boiling temperature liquids can generate gas; a property called vapor pressure. The molecules in the exterior layer of the liquid can escape into the air creating a vapor near the surface of the liquid, and this vapor exerts pressure. The warmer a liquid is, the more molecules can jump into the gas, and hence the vapor pressure increases. Liquids that evaporate at low temperatures are called *volatile* and have a high vapor pressure. In this experiment, we will create a gas by boiling a small amount of a volatile liquid.

Ideal gas law

In an ideal gas, particles do not interact at all. In real gases, particles do interact through attractive forces. A lot of gases act as an ideal gas at high temperatures and low pressures. On the other hand, the state of a gas is determined by a set of properties: pressure, volume, number of moles, and temperature. The volume (V) of a gas is the volume of its container, as gases occupy the whole container. Pressure (P), refers to the force that the gas particles exert on the walls of the container. Gas particles move fast and randomly, changing their direction after a collision with another particle or with the walls of the container. The stronger the collisions and the higher the frequency of collision, the higher the pressure. The number of moles of a gas (n), is the amount of particles. Finally, temperature (T) is related to the kinetic energy of the gas molecules. The higher the temperature the faster the average speed of the gas particles. All the variables that define the state of a gas are related through the ideal gas law:

$$\boxed{PV = nRT}\quad \text{Ideal Gas Law}$$

Pressure units

Gas pressure can be expressed in several units. Units of pressure are: atmospheres (atm), torr, pascals (Pa), millimeters of mercury (mmHg) and in inHg. In order to covert pressure units, you can use the following conversion factors:

$$\frac{1\ \text{atm}}{760\ \text{mmHg}} \qquad \frac{1\ \text{torr}}{1\ \text{mmHg}} \qquad \frac{1\ \text{atm}}{101325\ \text{Pa}} \qquad \frac{1\ \text{atm}}{29.92\ \text{inHg}}$$

The atmospheric pressure is normally close to one atm. However, its exact value depends on factors like altitude of even the weather. The higher you are with respect to the sea level the lower pressure. Similarly, the atmospheric pressure on a hot day is higher than on a cold day.

Example

The atmospheric pressure on a hot summer day in NYC is 29.84 inHg. Calculate this value in atm and mmHg.

Answer: In order to convert from inHg to atm we will use the conversion factor: 1 atm = 29.92 inHg and start by using the given pressure and the conversion factor with the inHg part on the bottom

$$29.84\ \cancel{\text{inHg}} \times \frac{1\ \text{atm}}{29.92\ \cancel{\text{inHg}}} = 0.9973\text{atm}$$

Now, we will convert this value into mmHg using 1 atm = 760 mmHg

$$0.9973\ \cancel{\text{atm}} \times \frac{760\ \text{mmHg}}{1\ \cancel{\text{atm}}} = 757.9\text{mmHg}$$

Calculating the molar weight of a volatile gas: the Dumas method

The procedure for calculating the molar weight (or molar mass) of a volatile gas–also called the Dumas method–consists of boiling completely a small amount of the liquid, so that the gas formed will occupy the volume of the container. In these conditions, you will know the volume of the gas (same as the container) and the temperature, which can be measured with a thermometer. The pressure of the gas will be the atmospheric pressure, as when a liquid boils its vapor pressure equals the external pressure. Using all these variables (P, V, T) we will be able to calculate the number of moles of the gas. At a given point we will stop heating the container and the vapor contained in the flask will condensate back into a liquid. Knowing the mass of the empty container and the mass of the container with condensate (the liquid) we will be able to measure the mass of the gas. Finally, the molar mass of a gas is the result of dividing its mass and its number of moles.

Example

We set up the experiment to calculate the molar mass of a volatile liquid. The mass of the dry flask is 90.00 g and after the experiment is done, the wet flask with the condensate liquid weighs 92.00 g. The volume of the flask is 150.0 mL. The temperature of the hot bath is 98.0°C and the atmospheric pressure is 0.98 atm. Calculate the molar mass of the substance.

Answer: To calculate the molar mass of the liquid we need the mass of the gas in the experiment and the number of moles. The mass of gas can be obtained from the difference in mass between the wet and dry beaker, as the condensate liquid comes from the gas in the flask, and hence the mass of this liquid is indeed the mass of gas in the experiment. The value is 2.00 grams. We will obtain the number of moles of gas using the ideal gas law. The pressure of the gas equals the atmospheric pressure. This is because when a liquid boils the vapor pressure equals to the atmospheric pressure: $P = 0.98\ atm$. On the other hand, the temperature of the gas is the temperature of the hot bath: $T = 98.0 + 273 = 371\ K$. The volume of the gas is the volume of the flask: $V = 0.150\ L$. Remember to use the ideal gas law we need to use atm, L, and Kelvins. Solve the ideal gas law for the n.

$$PV = nRT \qquad n = \frac{PV}{RT}$$

Plugging the values in the law:

$$n = \frac{0.98\,atm \cdot 0.150\,L}{0.082\,\frac{atm \cdot L}{mol \cdot K} \cdot 371\,K} = 4.83 \cdot 10^{-3}\,moles$$

Solving for the number of moles: $n = 4.83 \cdot 10^{-3}$ moles, and using the mass we can calculate the molar mass of the liquid:

$$MW = \frac{2.00\,\text{grams}}{4.83 \cdot 10^{-3}\,\text{moles}} = 414\,g \cdot mol^{-1}$$

Procedure

Generating the gas

☐ *Step 1:* – First, Prepare a hot water bath, using a 600 mL beaker, large enough to comfortably fit a 125 mL flask, and a hot plate. Add 200 mL of water to the beaker, place it on the hotplate, and start heating.

☐ *Step 2:* – Weight and record the mass of a dry 125 mL Erlenmeyer, covered with a small square of aluminum foil and fastened using a rubber band.

☐ *Step 3:* – The instructor will give you an unknown liquid. While you wait for the water to boil add approximately 2 mL of the unknown liquid into the Erlenmeyer flask.

☐ *Step 4:* – Place the aluminum foil in the mouth of the flask and fasten it with the rubber band. After that, make one very tinny hole on the aluminum foil cover, enough for excess vapor to escape while the liquid boils.

☐ *Step 5:* – When the water in the hot bath is boiling, place the Erlenmeyer flask inside the boiling water, close to the bottom of the beaker. Clamp the flask so that it does not move and twist slightly the flask so that you can see the level of the liquid in the flask. Wait until the unknown liquid has completely disappeared from the flask, becoming gas, and continue boiling the system for 10 minutes. At this point, you already have a gas inside the flask, at a certain temperature (that of the water) and pressure (the atmospheric pressure).

Measuring T and P

☐ *Step 6:* – While the gas is in the Erlenmeyer, measure its temperature by measuring the temperature of the boiling water. Record this measurement in the results and make sure you write down this value in Kelvins.

☐ *Step 7:* – Measure the pressure of the gas–remember this is equal to the atmospheric pressure–with a barometer in the lab. Record this measurement in the results, and make sure you write down these results in atmospheres.

☐ *Step 8:* – After 10 minutes, stop the hot plate and remove the beaker from the bath. Wait patiently until the flask is completely cool. At this point measure the mass of the flask, with the rubber band and aluminum foil and the condensed liquid. At this point, you will have the mass of the gas, which is the same as the weight of the condensed liquid.

Calculating the volume of an Erlenmeyer

☐ *Step 9:* – Calculate the volume of the Erlenmeyer by filling the flask with water, all the way to the brim. Use a graduated cylinder larger enough to accurately measure the volume of liquid in the flask. Help yourself with a plastic pipet to transfer all the water without spilling one drop. The volume should be more than 125 mL.

☐ *Step 10:* – Write down the volume of the Erlenmeyer in the results section, and make sure you write the result in liters. At this point, you will have the volume of gas.

☐ *Step 11:* – Use the ideal gas law to calculate the number of moles of gas. Use the mass measurement to calculate the molar mass of the unknown gas.

> ⚠ CAUTION!
>
> ⚠ Be aware that hot glass looks like cold glass.
>
> ⚠ All chemicals used must be disposed properly.

Calculations

① This is the mass measured using the balance.

② The measured temperature of the water boiling, measured using a thermometer.

③ Measured using the barometer.

④ This is the mass measured using the balance.

⑤ Calculate mass by subtraction: ④ – ①

⑥ Measured with a graduated cylinder.

⑦ Calculate using the Ideal Gas Law.

$$n = \frac{③ \cdot ⑥}{0.082 \frac{atm \cdot L}{mol \cdot K} \cdot ②}$$

⑧ This is the molecular weight of the mass:

$$MW = ⑤ / ⑦$$

STUDENT INFO

Name: Date:

Pre-lab Questions

Molar Mass of a Volatile Liquid

1. The atmospheric pressure of a hot summer day in NYC is 29.94 inHg. Calculate this value in mmHg and atm.

2. The atmospheric pressure of a cold winter day in NYC is 25.94 inHg. Calculate this value in Pa.

3. We set up an experiment utilizing the Dumas method to calculate the molar mass of an unknown chemical. A 4.00 mL pure liquid sample of this chemical is vaporized in an Erlenmeyer flask with a volume of 155 mL when the barometric pressure is 1.01 atm. The empty flask weighs 25.2436 g. After the excess gas escapes, the temperature is measured as 97.0°C. The flask and contents are subsequently cooled and the vapor condenses to a liquid. The weight of the flask and contents is found to be 25.7843 g. Calculate the molar mass of the unknown liquid.

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4. We heat a liquid until it boils. When it is boiling we leave the heat on. Does the temperature of the boiling liquid increase?

Results
EXPERIMENT

Molar Mass of a Volatile Liquid

Unknown # =

1	Mass of dry flask with foil and rubber (g)	
2	Temperature (K)	
3	Pressure (atm)	
4	Mass of wet flask with foil and rubber (g)	
5	Grams of gas (g)	
6	Volume (L)	
7	Moles of gas (mol)	
8	Molar Mass of the gas ($g \cdot mol^{-1}$)	

Post-lab Questions

Molar Mass of a Volatile Liquid

1. Dumas studied mercury intending to estimate its molar mass. He found that at 446 °C and 765 torr, 0.812 g of mercury vapor filled a vessel of volume 0.235186 L. From this data, compute the molar mass of mercury.

2. Calculate the mass of 45.0 L of NH_3 at 27 °C and 890 mmHg.

3. A volume of 30.0 mL of gas was collected in a tube at a temperature of 15°C and 800. mm Hg. The next day the volume of the same gas was 26.0 mL with the barometer still reading the same pressure. Calculate the temperature on the second day.

EXPERIMENT

Empirical rules for polarity and Paper Chromatography

Goal

This experiment will introduce you to paper chromatography, which is a standard chemical technique used to separate chemicals in a mixture. The goal of the experiment is to identify an unknown chemical in a mixture of several chemicals. You will do this employing paper chromatography.

Materials

- ☐ 4in tall × 9in long piece of filter paper
- ☐ 8 capillary tubes
- ☐ A 600 mL beaker

- ☐ A large watch glass to be used as a lid
- ☐ An unknown substance, some blanks solutions and a mixture solution.

Background

Often, the chemicals in nature exist as mixtures of several compounds and one needs to figure out the components. There are many analytical techniques used to separate and identify the components of a mixture. These techniques exploit the differences in the chemical or physical properties of the components of the mixture to separate the different elements. Chromatography is a very sensitive as well as affordable chemical technique, extensively employed to separate mixtures of chemicals and identify the nature of the elements of the mixture. This is a classic experiment, previously implemented in numerous college chemistry laboratory manuals with different reagents [1–4].

Mixtures

A lot of the materials that you use in your everyday life are indeed mixtures of chemicals. For example, air is a mixture of gases. Sometimes one needs to specify the composition of mixtures quantitatively, identifying each of the elements. In this experiment, we will learn to separate and identify the different compounds in a mixture.

Paper Chromatography

Paper chromatography relies on capillarity, which is the tendency of liquid substances to rise on the surface of a material. In this technique, a drop of a liquid solution containing different substances (the sample) is deposited on a rectangular piece of filter paper, close to the bottom edge. This paper is called the stationary phase. The bottom end of the paper is immersed in a liquid called the mobile phase, to a point that is just below the spot where the sample was placed. Due to capillarity, the mobile phase will move up along the stationary phase. When the mobile phase reaches the sample, the different components of the mixture will begin to migrate, carried away by the mobile phase. The chemical compounds forming the sample will move with the mobile phase, but as different chemicals have different tendencies to stick to the mobile phase, they will cover different distances in the stationary phase. The different heights achieved by the different substances would allow you to identify those chemicals. A component of the mixture with a high affinity to the mobile phase will migrate more than a component with a higher affinity to the stationary phase.

Retention factor, R_f

Each chemical will have a specific affinity towards the mobile phase, and as a consequence, it will move further in the stationary phase. The distance traveled by a component referred to as the distance traveled by the mobile phase in the experiment is a measure of the affinity between the chemical and the mobile phase. We define the retention factor R_f of a given chemical as:

$$R_f = \frac{\text{distance traveled by the chemical}}{\text{total distance traveled by mobile phase}}$$

The R_f value of a substance is characteristic of that substance. When dealing with mixtures one has to calculate the R_f for each pure component separately to then compare the retention factors with the ones obtained in the mixture.

How to reveal the spots

Most of the chemicals leave no mark when being carried by the mobile phase. For those cases, one needs to reveal the spots using a revealing agent. The revealing agent reacts with the components of the mixture giving a colored spot. The revealing is done after the paper chromatography is finished, to make the spots visible. Each spot is a chemical that was carried by the mobile phase a certain distance. If the sample is a pure chemical, you will see a single spot. If the sample is a mixture of chemicals you will see one spot for each component. Often the spots can overlap each other.

Empirical rules for polarity

The affinity between a chemical and the mobile phase is connected to a concept called polarity. Molecules contain electrons and depending on the electron distribution within the molecules, molecules can be polar or non-polar.
Molecules with an even electron distribution are non-polar. An example of this is H_2 molecule, which is non-polar. Differently, HF is a polar molecule, as F likes more the electrons than H and these will spend a longer time along fluorine (you will learn more about this effect at the end of the semester). The polar nature of substances is related to their miscibility and molecules with similar polar characteristics will mingle and mix creating a single visible phase. As an example, water (polar) and methanol (polar) will mix. Differently, water (polar) and oil (non-polar) are immiscible due to their different polar nature and they will not mix. Even if the rules or polarity are based on the nature and structure of the molecule, one can use very simple empirical rules to classify molecules as polar or non-polar. These rules work in general well for the case of diatomic and very large molecules:

 × Diatomic molecules made of the same element are non-polar.

 × Diatomic molecules made of different elements are polar.

 × Poliatomic molecules (with more than four atoms) made of C and H are in general non-polar.

 × Poliatomic molecules (with more than four atoms) containing C, H, and a different atom are in general polar.

Example

Classify the following molecules as polar or nonpolar: H_2, HCl, CH_3CH_3, and CH_3CH_2Cl.

Answer: H_2 is a non-polar molecule, being a diatomic molecule containing two atoms of the same element. Differently HCl is polar. CH_3CH_3 is a non-polar polyatomic molecule made of C and H atoms only, whereas CH_3CH_2Cl is polar.

References

(1)   Bergstrom, B. A.E.M. A., *Chemistry in Your Life Lab Manual*; W. H. Freeman: 2006.

(2)   Girard, P. B. J., *Laboratory Manual for Principles of General Chemistry*; Jones and Bartlett Learning: 2002.

(3)   Ebbing, R. W.D. D., *Experiments in General Chemistry*; Houghton Mifflin Harcourt Publishing Company: 2004.

(4)   Selfe, I. B.G.O. S., *General, Organic, and Biochemistry Lab Manual*; Freeman: 2006.

Procedure

Prepare the setup & add the chemicals

☐ *Step 1:* – Obtain the materials. Make sure the paper has the correct size, and if not you cut it.

☐ *Step 2:* – Take the piece of filter paper and use a pencil–never a pen–and a ruler to draw a line 2 cm from one of the longer edges. Draw another line 2 cm from the opposite edge.

☐ *Step 3:* – Fold the paper in half. Fold the paper again in half of the half. And once more before you unfold it, obtain an accordion-like flexure as shown in the figure below:
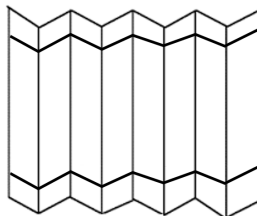
☐ *Step 4:* – You should now have the 2 pencil lines divided into 8 equal segments. If you are not sure about the result, show the folded paper to your instructor.

☐ *Step 5:* – Mark the center of each of the six inner segments of one line with an X (use only pencil), and write the labels: *A*, *B*, *C*, *D* and *U* under the marks as shown below. *A-C* are the blanks (references), *M* is a mixture of the four blanks and *U* is for the unknown.
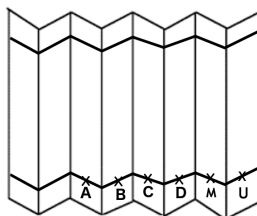
☐ *Step 6:* – With a clean capillarity tube, add a single drop of each chemical on its mark. Use a clean capillary for each blank so that the blanks do not mix. The drop should be small, barely visible, result of applying the capillary for 2 seconds on the filter paper.

☐ *Step 7:* – Now you will make the mobile phase by mixing about 30 mL of the acetone (or an equivalent organic solvent) with about 10 mL of concentrated HCl ⚠ solution in the 600 mL beaker. Cover the beaker with a watch glass so that the solvents do not evaporate.

> **Good Lab Practice**
>
> Obtain directions from your instructor before discharging the chemicals in this experiment. ✎

Starting the chromatography

☐ *Step 8:* – Very gently, place the folded paper standing inside the beaker with the X-marked line in the lower part. The liquid should not reach the line with the marks (see image below). Cover the beaker with the watch glass again. Make sure the paper is not coming out of the beaker. If the paper touches the lit of the walls of the beaker you will have to repeat all the steps above.

☐ *Step 9:* – Once the chromatography has started you are not allowed to move either the beaker or the paper until the experiment is finished.
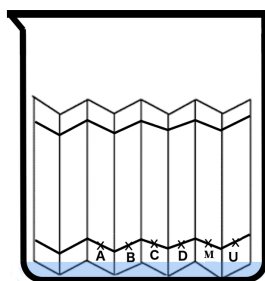
Good Lab Practice

You are not allowed to move the beaker after the chromatography has started

☐ *Step 10:* – The accordion paper should be flat on the liquid and the beaker must be stationary throughout the experiment.

☐ *Step 11:* – Now the mobile phase will move up passing by the bottom line. Make sure you see the line of the wet paper moving upwards.

☐ *Step 12:* – When the liquid has reached the top line open the beaker and take out the paper.

Revealing the marks

☐ *Step 13:* – This part should be done in the hoods and you will only take the paper out when it is completely dry.

☐ *Step 14:* – Wearing plastic gloves, spray the paper with a solution of NH_3 ⚠. The paper should never be wet but just moist.

☐ *Step 15:* – Spray the paper with a solution of NaS_2 ⚠.

☐ *Step 16:* – Let the the paper dry in an adjacent hood (you can use a hair dryer to speed up the process) and only when it is totally dry you can return it to your station.

☐ *Step 17:* – Make sure the paper does not leave the hood until is completely dry.

☐ *Step 18:* – Circle each spot with a pencil.

☐ *Step 19:* – Measure the vertical distance that the approximate center of each of these spots has traveled from the bottom 2-cm-line. Record the distances in the Results table.

☐ *Step 20:* – Measure the vertical distance traveled by the mobile phase (the front of the liquid) from the bottom 2-cm-line. Record the distance in the Results table.

☐ *Step 21:* – Calculate the R_f value for each spot.

☐ *Step 22:* – Use the R_f values to identify each component in the unknown mixture.

☐ *Step 23:* – Dispose ✍ the different chemicals in the waste disposal. Make sure you give the unknown back to your instructor.

> ⚠ CAUTION!
>
> ⚠ Sodium sulfide (NaS_2) is very toxic and should never be tasted or inhaled.
>
> ⚠ Nitric acid (HNO_3) and Ammonia (NH_3) can cause chemical burns.

Calculations

① Measure the vertical distance that the approximate center of each of these spots has traveled from the bottom 2-cm-line.

② Measure the vertical distance travelled by the mobile phase (the front of the liquid) from the bottom 2-cm-line.

③ You obtain this result by dividing:

$$② / ①$$

STUDENT INFO

Name: Date:

Pre-lab Questions

Empirical rules for polarity and Paper Chromatography

1. Define mobile and stationary phase.

2. A pair of chemicals are separated using paper chromatography using methanol (CH_3OH) as the carrier solvent. The chemicals that were separated are ethanol (CH_3CH_2OH) and Benzene (C_6H_6). Which substance will have the larger R_f value? Explain your reasoning.

3. The figure below reports a chromatogram for the separation of three compounds (I, II, and III), where the solvent level is marked with a dotted line. Calculate R_f for each compound.
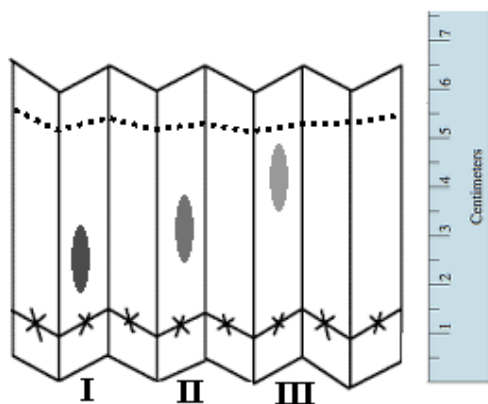
	I	II	III
R_f			

Results
EXPERIMENT

Empirical rules for polarity and Paper Chromatography

---

(1) Distance traveled by the mobile phase (cm): _____

	Blank name	Distance travelled (cm) (2)	R_f values (3)
A			
B			
C			
D			
Unknown	use as many rows as spots reveal in the unknown sample		

Components of the unknown mixture: _____

Post-lab Questions

Empirical rules for polarity and Paper Chromatography

1. For the following experiment, identify the chemicals in the unknown and calculate R_f for each compound.

EXPERIMENT

Solubilities Within a Family: Periodic properties of elements and compounds

Goal

The goal of this experiment is to qualitatively look for *atomic trends* in a series of atoms and molecules. On one hand, you will quan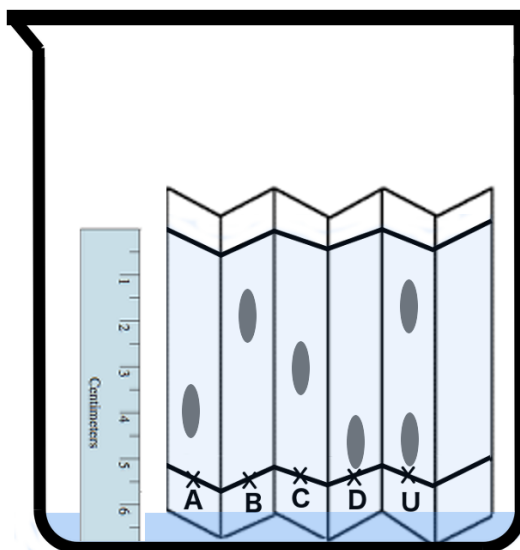titatively study the change of several atomic properties such as the atomic radius, the ionization energy, and electron affinity on the periodic table. On the other hand, you will compare the solubility of a series of compounds across the periodic table. Overall, this study demonstrates the different trends that atomic and molecule properties follow in the periodic table.

Materials

On one hand, this is a modeling-based experiment, and you will need internet connection and a computer to complete the first part of the experiment. All properties in this experiment will be taken from ♟ *www.webelements.com*. On one hand, you will need a series of reagents and materials for the second part of the experiment:

☐ A 5 mL graduated cylinder (or 10 mL)

☐ 5 small test tubes

☐ the following solutions: (a) 0.1M $Mg(NO_3)_2$, $Ca(NO_3)_2$,

$Sr(NO_3)_2$, $Ba(NO_3)_2$, and $Pb(NO_3)_2$

☐ the following solutions: (a) 0.1M NaOH, NaCl, NaBr, NaI, Na_2SO_4, Na_2CO_3, and $Na_2C_2O_4$

Background

The periodic table can be used to estimate atomic properties, as the elements in the table are arranged in order of increasing atomic number (Z). At the same time, the elements are arranged in such a way that the electron configuration displays a periodic variation: an equivalent kind of electron configuration occurs within a group (a vertical column) and varies along each period (horizontal row) on the table. For example, F and Cl are in the same group and their electronic configuration is $[He]2s^2 2p^5$ and $[Ne]3s^2 3p^5$, respectively. while H and He are located in the same period (period 1) and their configuration is $1s^1$ and $1s^2$, respectively.

The Periodicity of Atomic Properties

Atoms do not have well-defined boundaries because the electrons in the electronic cloud are delocalized in space. Still, when they form solids or molecules, the distance between atoms can be measured. This measurement allows us to define an *atomic radius* as half the distance between the neighboring atoms. Atoms can lose or gain electrons forming cations or anions, respectively. The radius of cations and anions–*the ionic radius*–differ from the radii of their parent atoms. On one hand, all cations are smaller than their parent atoms, whereas anions are larger than their parent atoms.

The *ionization energy, I*, is the energy needed to remove an electron from a neutral atom in the gas phase. Elements with low ionization energies can easily form cations, whereas elements with high ionization energies are unlikely to form cations. I is normally expressed in electron-volts (eV), a unit that is related to joules by the following conversion factor:

$$\frac{1\,eV}{1.602 \times 10^{-19}\,J}$$

Sometimes I is expressed in kJ/mol that can also be converted to eV:

$$\frac{1\,kJ/mol}{1.04 \times 10^{-2}\,eV}$$

The *electron affinity*, EA, of an element is the energy released when a single electron is added to a gas-phase atom. Positive electron affinities mean that energy is released when an electron is added to an atom, whereas negative EA values mean that energy must be supplied to attach an electron to an atom. Similar to ionization energies, EA values are reported in eV for a single atom. All atomic properties–atomic radius, ionization energy, and electron affinity–can be found on web-based periodic tables such as ✺ www.WebElements.com.

Example

Find the values of the atomic radius (in pm), the first ionization energy, and the electron affinity (in eV) for Chlorine in ✺ www.WebElements.com (1 pm = $1 \cdot 10^{-12}$ m).

- -

Answer: Look for the Chlorine section in www.WebElements.com, and click on the 'More Properties → Atoms Sizes' menu, you will find the 'empirical' atomic radius, the one calculated experimentally, with a value of $100\,pm$. To find the first ionization energy of Cl, navigate to the 'More Properties → Electron shell data' menu. It is $1251.2\,kJ/mol$. We can convert this value to eV:

$$1251.2\,kJ/mol \times \frac{1.04 \times 10^{-2}\,eV}{1\,kJ/mol} = 13.01\,eV$$

The electron affinity of Cl is 349 kJ/mol which is 3.6 eV.

Finally, the electronegativity of an element i is a measure of the tendency of an atom to attract a bonding pair of electrons. The Pauling scale is the most commonly used.

## The Periodicity of Molecular Properties

Mendeleev's periodic table of atoms is one of the most important principles in natural science. Even if there is not such a thing for molecules, one can arrange some molecular properties according to the periodic table. For example, HF is a covalent molecule with a molecular H-F distance of 92pm. The bond distance for HCl, HBr and HI is 127, 141, and 161 pm, respectively. These results show that for the HX distance, with X standing for F, Cl, Br, and I, the H-X molecular distance increases as we decrease in the group. In a similar way the molecular bond of BeH_3 is 119pm, whereas the same parameter for CH_4, NH_3, H_2O and HF is 109, 96 and 92 pm. These results indicate that the molecular H-X bond decreases when going from left to right of a period.

Solubility and double replacement reactions

Learn more about solubility in this ✺ Video Link

Visual

Precipitation reactions occur when dissolved substances react to form a solid product. Many reactions of this type involve the exchange of ions between ionic compounds in an aqueous solution and are sometimes referred to as double-replacement reactions. For example, alkaline earth metals and lead form nitrates, hydroxides, chlorides, bromides, and iodides with the general formulas $M(NO_3)_2$, $M(OH)_2$, MCl_2, MBr_2 and MI_2, respectively, where M stands for the alkaline earth and lead cation. These compounds react with sodium hydroxides, chlorides, bromides, and iodides in water through the reaction:

$$M(NO_3)_2 \quad + \quad 2\,NaX \quad \longrightarrow \quad MX_2 \quad + \quad 2\,NaNO_3$$
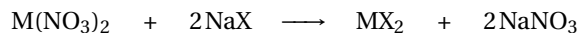
where X stands for hydroxide (OH^-), chloride (Cl^-), bromide (Br^-), or iodide (I^-). In this set of reactions, M replaces Na in NaX, and NO_3 replaces X in NaX, producing a double replacement. $NaNO_3$ is soluble in water and hence if a precipitate appears it can only be due to MX_2. We will use this trick to determine the qualitative solubility of the hydroxides, chlorides, bromides, and iodides in water.

In a similar way, alkaline earth metals and lead also form sulfates, carbonates, oxalates, and chromates with the general formula MSO_4, MCO_3, MC_2O_4, and $MCrO_4$, respectively, where M stands for the alkaline earth and lead cation. These compounds react with sodium hydroxides, chlorides, bromides, and iodides in water through the reaction:

$$M(NO_3)_2 \quad + \quad Na_2Y \quad \longrightarrow \quad MY \quad + \quad 2\,NaNO_3$$
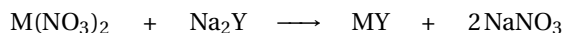
where Y represents sulfate, carbonate, oxalate, and chromate. The solubility of the MY compounds can be determined in the same way as those of the MX compounds.

Procedure

Periodic properties of atoms

☐ *Step 1:* – Record from ✆ www.WebElements.com the atomic radius, first ionization energy, the electron affinity, and the Pauli's electronegativity of the elements in the *Results* sections. Convert the energy units to eV.

☐ *Step 2:* – Plot the atomic radius, the first ionization energy in eV, and the electron affinity in eV vs. the atomic number in the graph paper provided.

☐ *Step 3:* – Make sure you include all elements in the plot, connecting the dots with lines and using labels to identify the name of the element in the plot.

Periodic properties of compounds

☐ *Step 1:* – Obtain 5 small test tubes, marking them with *identification numbers*.

☐ *Step 2:* – In the following steps you will create several *1mL marks* on the test tubes. To do this: first use a 5mL (or 10mL) graduated cylinder to place 1mL of distilled water in each of these test tubes. Mark the height of the water in each test tube with a small piece of tape or marking pencil.

☐ *Step 3:* – Now add 1mL to each test tube and mark the new height of the water in each.

☐ *Step 4:* – After creating all marks, pour the water into the sink.

> **Good Lab Practice**
>
> ✍ Obtain direction from your instructor for discharging the solutions ✍

Determining qualitatively the solubility of a set of hydroxides X(OH)$_2$

☐ *Step 1:* – Using the lower marks on the test tube as a guide, add 1mL of 0.1M $Mg(NO_3)_2$ to the first test tube. Then add 1mL of 0.1 M $Ca(NO_3)_2$ to the second, 1mL of 0.1M $Sr(NO_3)_2$ to the third, 1mL of 0.1M $Ba(NO_3)_2$ to the fourth and 1mL of 0.1M $Pb(NO_3)_2$ to the fifth test tube. Make sure you are aware of the color and clarity of each solution.

☐ *Step 2:* – Using the upper marks as guides, add 1mL of 1M NaOH to each test tube. Shake each tube gently and wait for about 30s.

☐ *Step 3:* – Notice the colors of all precipitates. A precipitate may come across as a change in the clarity of the solution. A precipitate may appear as 'cloudy' or milky. Record your observations in the results table.

☐ *Step 4:* – Discard the contents of each of the test tubes as directed by your laboratory instructor. Wash the test tubes carefully and rinse them with distilled water.

> ⚠ CAUTION!
>
> ⚠ Lead, Barium, and oxalate are toxic. Mind washing your hands thoroughly after using those compounds.
>
> !

Determining qualitatively the solubility of a set of chlorides (XCl_2), bromides (XBr_2) and iodides (XI_2)

☐ *Step 1:* – Using the lower marks on the test tube as a guide, add 1mL of 0.1M $Mg(NO_3)_2$ to the first test tube. Then add 1mL of 0.1 M $Ca(NO_3)_2$ to the second, 1mL of 0.1M $Sr(NO_3)_2$ to the third, 1mL of 0.1M $Ba(NO_3)_2$ to the fourth and 1mL of 0.1M $Pb(NO_3)_2$ to the fifth test tube. Make sure you are aware of the color and clarity of each solution.

☐ *Step 2:* – Using the upper marks as guides, add 1mL of 1M NaCl to each test tube. Shake each tube gently and wait for about 30s.

☐ *Step 3:* – Notice the colors of all precipitates. A precipitate may come across as a change in the clarity of the solution. A precipitate may appear as 'cloudy' or milky. Record your observations in the results table.

☐ *Step 4:* – Discard the contents of each of the test tubes as directed by your laboratory instructor. Wash the test tubes carefully and rinse them with distilled water.

☐ *Step 5:* – Repeat the previous steps six more times in turn adding each of the following solutions instead of the solution of NaCl: 0.1M NaBr and 0.1M NaI.

Determining qualitatively the solubility of a set of sulfates ($Na(SO_4)_2$), carbonates ($Na(CO_3)_2$) and oxalates ($Na_2C_2O_4$).

☐ *Step 1:* – Using the lower marks on the test tube as guides, add 1mL of 0.1M $Mg(NO_3)_2$ to the first test tube. Then add 1mL of 0.1 M $Ca(NO_3)_2$ to the second, 1mL of 0.1M $Sr(NO_3)_2$ to the third, 1mL of 0.1M $Ba(NO_3)_2$ to the fourth and 1mL of 0.1M $Pb(NO_3)_2$ to the fifth test tube. Make sure you are aware of the color and clarity of each solution.

☐ *Step 2:* – Using the upper marks as guides, add 1mL of 0.1M $Na(SO_4)_2$ to each test tube. Shake each tube gently and wait for about 30s.

☐ *Step 3:* – Notice the colors of all precipitates. A precipitate may come across as a change in the clarity of the solution. A precipitate may appear as 'cloudy' or milky. Record your observations in the results table.

☐ *Step 4:* – Discard the contents of each of the test tubes as directed by your laboratory instructor. Wash the test tubes carefully and rinse them with distilled water.

☐ *Step 5:* – Repeat the previous steps six more times in turn adding each of the following solution instead of the solution of $Na(SO_4)_2$: 0.1M $Na(CO_3)_2$, and 0.1M $Na_2C_2O_4$.

Calculations

① In ♣ www.WebElements.com for each element, click on the 'Atoms Sizes' menu, you will find the 'empirical' atomic radius R, the one calculated experimentally. Write this value on the *Results* table.
Plot R vs. the atomic number Z. R, in pm, goes in the vertical axis, whereas Z goes in the horizontal axis.

② In ♣ www.WebElements.com for each element, you will find the 'first ionization energy' I in kJ/mol. Write this value on the *Results* table.

③ To convert the ionization energy I from kJ/mol to eV do:

$$② \times \frac{1.04 \times 10^{-2}\, eV}{1\, kJ/mol}$$

Plot I vs. the atomic number Z. I in eV goes in the vertical axis, whereas Z goes in the horizontal axis.

④ In ✿ www.WebElements.com for each element, selecting 'all properties' you will find the electron affinity EA in kJ/mol. Write this value on the *Results* table.

⑤ To convert the electron affinity EA from kJ/mol to eV do:

$$④ \times \frac{1.04 \times 10^{-2} \, eV}{1 \, kJ/mol}$$

Plot EA vs. the atomic number Z. EA in eV goes in the vertical axis, whereas Z goes in the horizontal axis.

Pre-lab Questions

Solubilities Within a Family: Periodic properties of elements and compounds

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1. The first ionization potential of Carbon is 1086.45 kJ/mol. Convert this value into eV.

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2. The atomic radius of Carbon is 70pm. Convert this value into meters.

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3. Using ✤ www.WebElements.com fill the following table with the properties of the atom indicated.

    This table in the pdf is writable only with Acrobat Reader...

Element	Atomic Number Z	Atomic radius (pm)	First Ionization energy		Electron affinity	
			(kJ/mol)	(eV)	(kJ/mol)	(eV)
H						

4. Using ✤ www.WebElements.com fill the following table with the properties of the atom indicated.

    This table in the pdf is writable only with Acrobat Reader...

Element	Atomic Number Z	Atomic radius (pm)	Electronegativity
Li			

5. Name or give the formula for the following compounds:

$Mg(NO_3)_2$	_____	Sodium chloride	_____
$Ca(NO_3)_2$	_____	Sodium bromide	_____
$Sr(NO_3)_2$	_____	Na_2SO_4	_____
$Ba(NO_3)_2$	_____	Na_2CO_3	_____
$Pb(NO_3)_2$	_____	$Na_2C_2O_4$	_____

6. Give the formula for the compound resulting of mixing the following chemicals:

$Mg(NO_3)_2 + NaOH$	_____	$Ca(NO_3)_2 + Na_2CO_3$	_____
$Mg(NO_3)_2 + NaCl$	_____	$Ca(NO_3)_2 + Na_2C_2O_4$	_____
$Mg(NO_3)_2 + NaBr$	_____	$Sr(NO_3)_2 + NaOH$	_____
$Mg(NO_3)_2 + NaI$	_____	$Ba(NO_3)_2 + NaI$	_____
$Mg(NO_3)_2 + Na_2SO_4$	_____	$Pb(NO_3)_2 + Na_2SO_4$	_____

Solubilities Within a Family:
Periodic properties of elements

STUDENT INFO

Name: _____   Date: _____

Results

EXPERIMENT

Element	Atomic Number Z	Atomic radius (pm) ①	First Ionization energy (kJ/mol) ②	(eV) ③	Electron affinity (kJ/mol) ④	(eV) ⑤	Electronegativity
H							
He							
Li							
Be							
B							
C							
N							

Element	Atomic Number Z	Atomic radius (pm) ①	First Ionization energy (kJ/mol) ②	First Ionization energy (eV) ③	Electron affinity (kJ/mol) ④	Electron affinity (eV) ⑤	Electronegativity
O							
F							
Ne							
Na							
Mg							
Al							

Element	Atomic Number Z	Atomic radius (pm) ①	First Ionization energy (kJ/mol) ②	First Ionization energy (eV) ③	Electron affinity (kJ/mol) ④	Electron affinity (eV) ⑤	Electronegativity
Si							
P							
S							
Cl							
Ar							
K							

Element	Atomic Number	Atomic radius	First Ionization energy		Electron affinity		Electronegativity
	Z	(pm) ①	(kJ/mol) ②	(eV) ③	(kJ/mol) ④	(eV) ⑤	
Ca							
Ga							
Ge							
As							
Se							
Br							
Kr							

Figure 13: Atomic Radius Plot: Atomic radius (Y), atomic number(X)
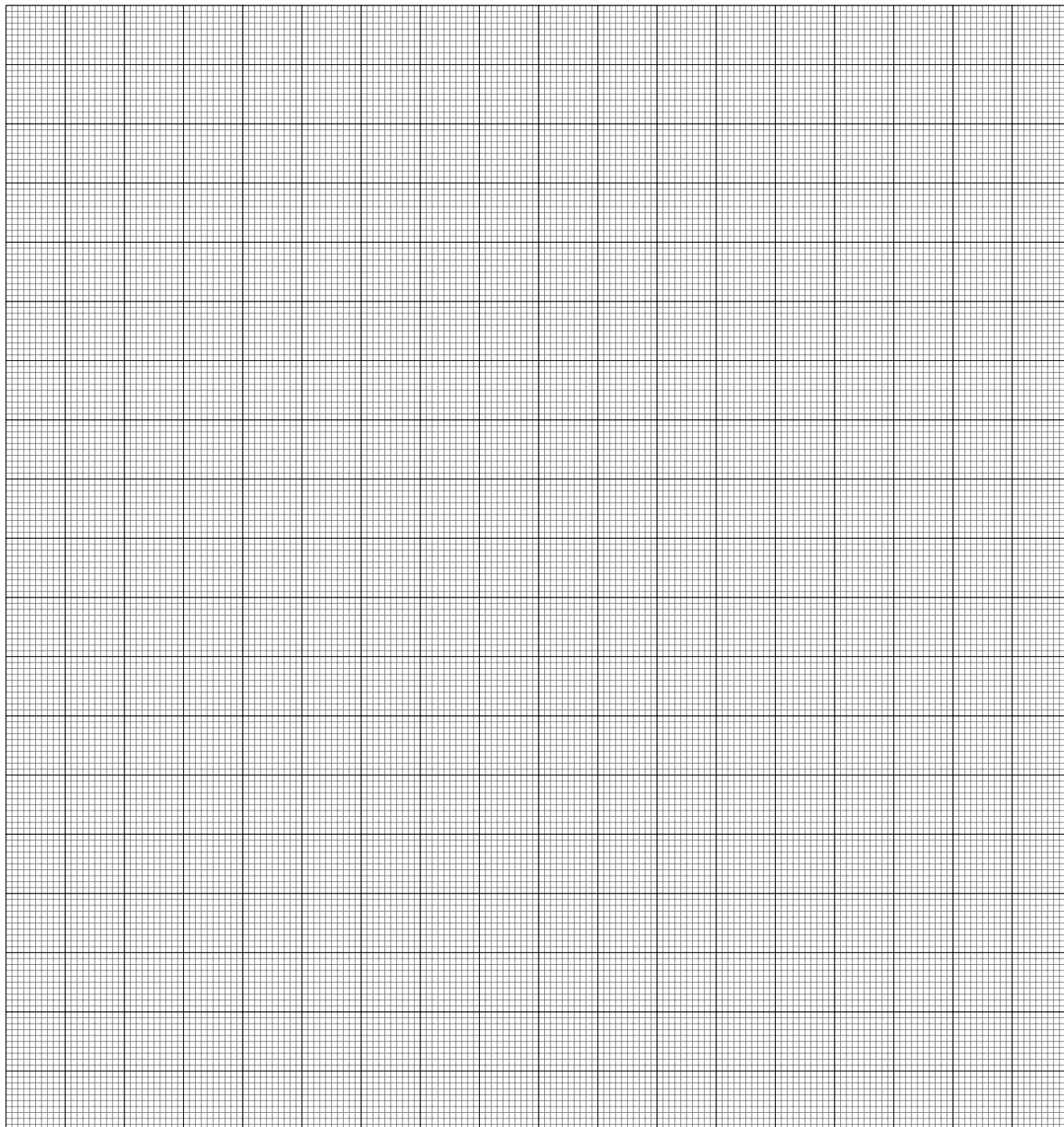
Figure 14: Ionization Potential Plot: Ionization potential (Y), atomic number(X)
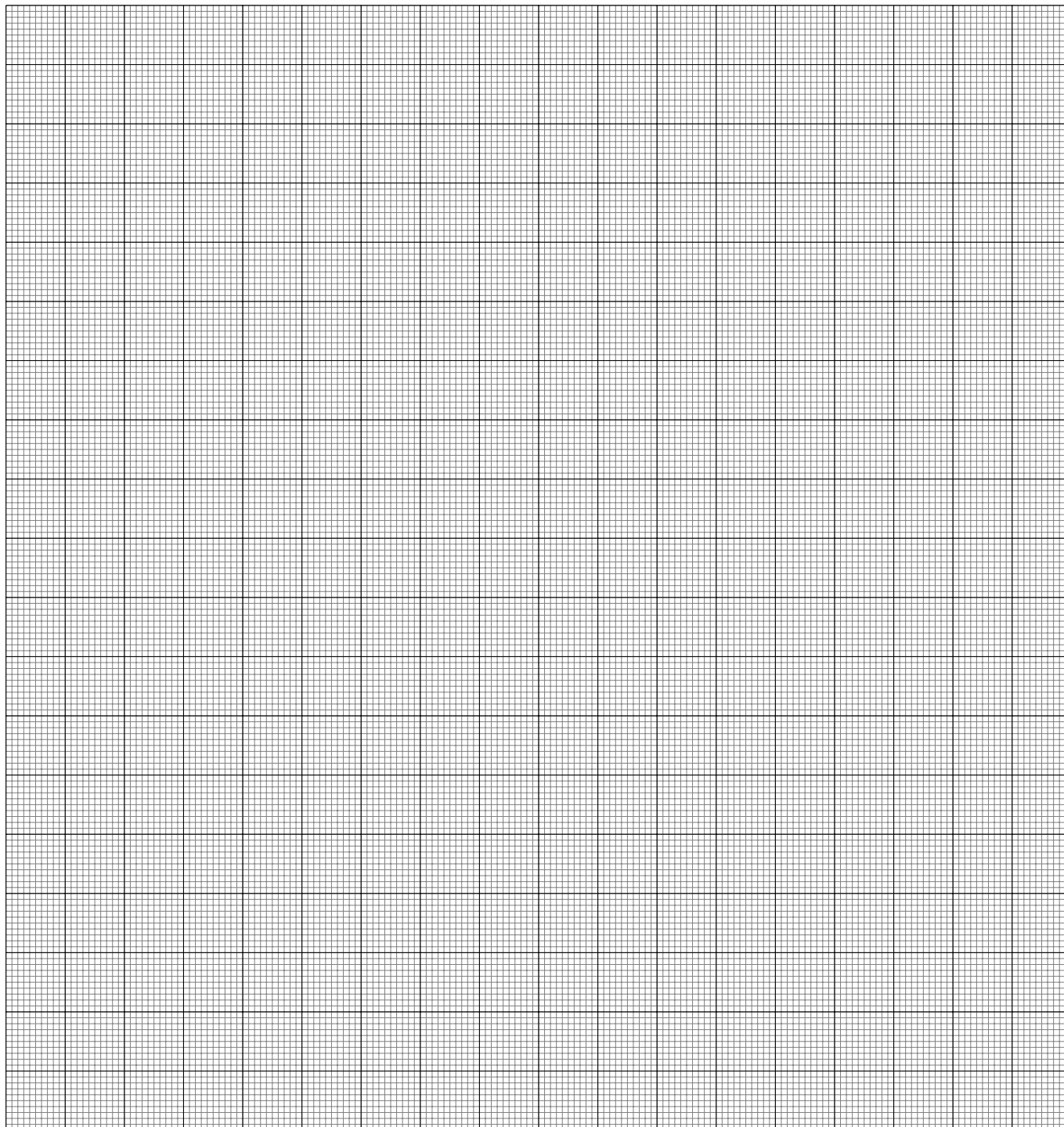
Figure 15: Electron Affinity Plot: electron affinity (Y), atomic number(X)
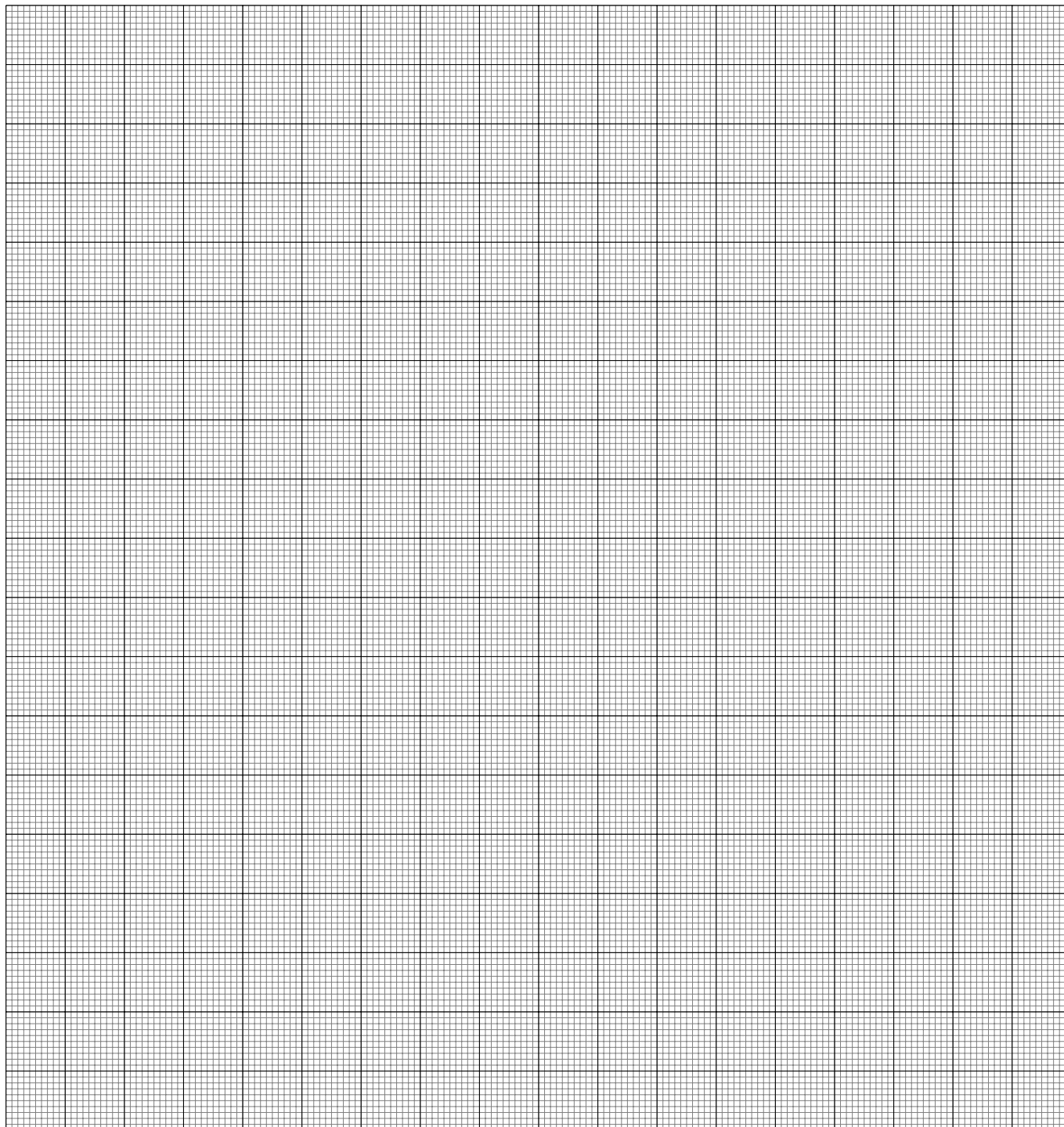
Figure 16: Electronegativity: electronegativity (Y), atomic number(X)

Periodic properties of compounds

	Mg(NO$_3$)$_2$	Ca(NO$_3$)$_2$	Sr(NO$_3$)$_2$	Ba(NO$_3$)$_2$	Pb(NO$_3$)$_2$
NaOH					
NaCl					
NaBr					
NaI					
Na$_2$SO$_4$					
Na$_2$CO$_3$					
Na$_2$C$_2$O$_4$					

(Write *P* for precipitate, *SP* for slighly precipitate, *BP* for barely precipitate, and *NP* for no precipitate. If insoluble, indicate the color of the precipitate.)

Post-lab Questions

Solubilities Within a Family: Periodic properties of elements and compounds

1. From your data, indicate how the atomic radius changes across a period and across a row of the periodic table.

2. From your data, indicate how the ionization energy changes across a period and across a row of the periodic table.

3. From your data, indicate how the electron affinity changes across a period and across a row of the periodic table.

4. From your data, indicate how the electronegativity changes across a period and across a row of the periodic table.

5. What can you conclude from your data about the trends in the solubility of the halide compounds as the atomic number increases?

6. What can you conclude from your data about the trends in the solubility of the hydroxides as the atomic number increases?

7. What can you conclude from your data about the trends in the solubility of the sulfates, carbonates and oxalates as the atomic number increases?

8. Compare the solubility of the lead compounds and the alkali earth metals. Give and example while explaining these differences.

<div align="center">

EXPERIMENT

Molecular Geometry

</div>

Goal

This experiment will go over the ideas of molecular geometry and bond-hybridization. On one hand, you will learn how to predict the geometry of a molecule and how to differentiate, for example, a linear molecule from a bent molecule. Also you will learn to predict the hybridization of atomic orbitals involved in a chemical bond.

Materials

This is a theory-based experiment. You will not carry any lab work during this experiment and you only need a molecular models kit if available.

Background

Molecules result from the combination of atoms. For example, a H_2O molecule results of the combination of one oxygen atom with two hydrogen atoms. The atoms of a molecule combine by exchanging or sharing electrons, depending on the type of bond. Water is a covalent molecule and the oxygen and hydrogen atoms share the electrons in the bond. Only electrons in the outermost electron shell participate in chemical bonds. Those electrons are called valence electrons. Core electrons, electrons in inner shells of the atom, do not participate in chemical bonds. The electron configuration of oxygen is $1s^2 2s^2 2p^4$. Valence electrons are highlighted with bold letters and are those in the energy level $n = 2$. Chemists use several theories to describe the formation of a bond, and here we will cover three of these theories: the valence-shell electron-pair repulsion model (VSEPR), the valence bond theory, and the molecular orbital theory. These theories describe different aspects of the formation of a bond, sometimes complementing each other.

Valence Electrons

The valence electrons of an atom are those located in the electronic valence shell. For example, the electron configuration of lithium is $1s^2 2s^1$. This atom has two electrons in the $1s$ atomic orbital and one electron in the $2s$ higher-energy atomic orbital. The first two electrons belongs to the core, as the level $1s$ is completely filled with electrons. Differently, the second energy level is not completely filled and hence the single electron in this level is a valence electron. Another example would be oxygen, $1s^2 2s^2 2p^4$, which contains two core electrons–located in the $1s$ level–and six valence electrons. The number of valence electrons of an atom can be easily determine by looking at the group number. For example, oxygen belongs to the group 16(6A) and has six valence electrons. Similarly, nitrogen belongs to group 15(5A) and hence will have five valence electrons.

The Octet rule

Atoms gain or loose electrons when they combine to form molecules. The octet rule says that each atom in a molecule is surrounded by eight electrons. There are two important exceptions to this rule: hydrogen (H) will only surrounded by two electrons, and boron (B) by six.

Lewis dot symbol of an atom

The Lewis dot symbol of an atom is a representation of the element symbol surrounded by the valence electrons in the form of dots. For example, Li has one valence electron and hence its Lewis dot symbol will be ·Li . The Lewis dot symbol for O, with six valence electrons, will be ·Ö·. At this point is not that important how do you distribute the dots, and in case of doubt a wise choice is to pair the dots.

Lewis structure of diatomic molecules

In order to build up Lewis structures or electron-dot structures for diatomic molecules,

1. set up the element symbols next to each other.

2. count the total number of valence electrons in the molecule, by adding the valence electrons of each atom.

3. Add as many electrons (dots) as necessary to complete the octets (or duets) for each element. Make sure that there are at least two electrons shared (a single bond) between the two elements.

4. Count how many electrons are used in the resulting structure

   (a) If the structure uses as many electrons as valence electrons are available, this is the final structure.

   (b) If the structure uses more electrons than valence electrons are available, double bonds (four electrons shared) or triple bonds (six electrons shared) need to be used. Each double bound will save two electrons with respect to a single bond. Each triple bound will save two electrons with respect to a double bound.

Lewis structures of polyatomic molecules

For polyatomic molecules the procedure is exactly the same. The only difference is to arrange the elements correctly. Typically, finding the central atom is the key. Two tricks to find the central atom.

1. the central atom is the one with a lower index in the molecule (e.g. in H_2O is O or in NH_3 is N)

2. the central atom is that with the lowest electronegativity.

The pairs of electrons that connect two atoms are called *bonds*. In Lewis structures these two electrons can be replaced by lines joining the elements. The pairs not involved in a bond are called *lone pairs*. Notice that the atoms arrangement (if the structure looks like a line, a triangle or so) is not necessary representative of the real, three-dimensional molecular geometry.

Example

Construct the electron-dot structure of H_2O indicating the number of bonds and lone pairs.

- -

Answer: we first arrange the atoms in the molecule as H O H. The central atom is O, as oxygen has the lower index in the H_2O molecule–the index for O is one and the index for H is two. Now we count the total number of valence electrons, including all atoms: $2xH(1e^-)$ and $O(6e^-)$ that gives a total of eight electrons. Now we distribute the pair on each atoms knowing that each atom has to have 8 electrons with the exception of hydrogen that can only be surrounded by two.

$$H:\ddot{O}:H$$

and using lines instead of pairs (this is not necessary but makes the electron-dot structure look better) we obtain

$$H\!-\!\ddot{O}\!-\!H$$
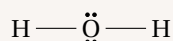
The molecule has two bonds, each one connecting a H to the oxygen atom, and two lone pairs located on the oxygen atom.

Atomic charges in a molecule and polyatomic ions

In order to build up the electron-dot structures of a molecule you need to count the number of valence electrons of the whole molecule. Each atom contributes with a different number of valence electrons to the molecule. For example, H contributes with one electron whereas O contributes with two. When you arrange the electron pairs in the molecule, each atom should have no less than the number of electrons that they bring. For example in the electron-dot structure of HCl, H:Cl: the hydrogen atom contributes with one electron to the molecule, and in the molecule the H atom owns one electron, as in H: one of the dots belongs to H and the other belongs to the Cl–the electrons are shared in a covalent bond. In the same way, the Cl atom contributes with seven electrons and in the molecule it owns seven electrons, as in :Cl: one of the dots belongs to H and the other seven belong to Cl. In another words, the H:Cl: electron-dot structure is the combination of H· and ·Cl:. We say that the charges on each atom are zero, as each atom in the molecule owns the same number of electrons that it originally brings. The lewis structure should also disp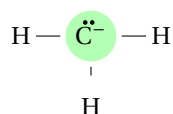lay the electron distribution in polyatomic ions. Look for example at CH_3^- ions. Comparing the valence electrons of each atom and the number of electrons surrounding each atom in this structure, one can see that the C atom has an extra electron, and hence it is responsible of the negative charge:

$$H - \overset{\cdot\cdot}{C}{}^- - H$$
$$|$$
$$H$$

VSEPR theory and molecular geometry

Molecules are arrangements of atoms, and these arrangements can have different forms. Think about a H_2O molecule, which contains two hydrogen atoms and one oxygen. Knowing that both hydrogens are connected to oxygen by means of a covalent bond, one can envision several molecular geometries such as

$$H - \overset{\cdot\cdot}{\underset{\cdot\cdot}{O}} - H \quad \text{or} \qquad \text{maybe} \qquad \overset{\cdot\cdot}{O}\overset{\cdot\cdot}{} \\ H \qquad H$$
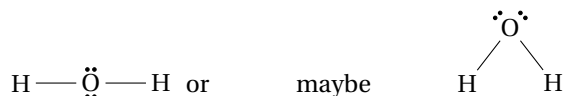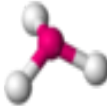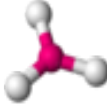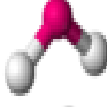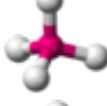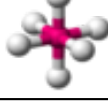
The goal of this section is to identify the geometry of a given molecule. In order to do this, the electron-dot structure of the molecule is the key. If the molecule contains two atoms, there is only a possible geometry these two atoms can exhibit, and this is a linear arrangement. For the case of more complex molecules, in order to identify the geometry you need to figure out the ABE code of the molecule. In this code B refers to the number of atoms connected to the central atom in a molecule, and E is the number of lone pairs on the central atom. For example, the electron-dot $H - \overset{\cdot\cdot}{\underset{\cdot\cdot}{O}} - H$ structure has two bonds with the central atom B_2 and two lone pairs on top of the central atom E_2 and hence the ABE code of the molecule would be AB_2E_2. Another example, the ABE code for ammonia

$$H - \overset{\cdot\cdot}{N} - H$$
$$|$$
$$H$$
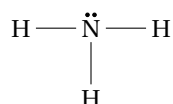
would be AB_3E, as the molecule has three atoms connected to the central nitrogen and and N has a single lone pair. You can find a list of the equivalence between ABE codes and the molecular geometry in Table 1. In order to predict the geometry of a molecule, once you have the ABE code, Table 1 will give you the geometry. For example, an AB_2 molecule will be linear, whereas an AB_2E_2 is a bent. The bond angles are also indicated in the table, and for example a CO_2 molecule, which will be linear, will have 180° angles. This means both C-O bonds will form a line. The *number of electron regions* is often referred as the result of adding the E and the B in the ABE code. For example, a AB_3E_2 molecules has five electron regions.

Table 4 Molecular geometries			
ABE Code	Molecular shape	Bond Angle	3D model
AB_2	Linear	180°	
AB_3E	Trigonal pyramidal	109°	
AB_3	Trigonal Planar	120°	
AB_2E_2	Bent	109°	
AB_2E	Bent	120°	
AB_5	Trigonal bipyramidal	90°, 120°,180°	
AB_4	Tetrahedral	109°	
AB_6	Octahedral	90°, 180°,180°	

Atomic orbitals

There are four main types of atomic orbitals: s, p, d and f. Each orbital type has a specific symmetry. For example, s orbitals are spherical, whereas the p orbitals consist of two lobes on opposite sides of the nucleus.

Valence bond theory and hybrid orbitals

The *valence bond theory* is a qualitative model that describes the formation of the chemical bonds. It assumes that the electrons of a molecule are located in atomic orbitals, as if the atoms in the molecule where separate from each other. These valence orbitals combine to produce mixed orbitals, known as *hybrid orbitals*. The mixing of orbitals is called *orbital hybridization*. Examples for orbital hybridization are sp, sp^2, sp^3 or sp^3d. In the previous examples, s and p orbitals are mixed giving sp, sp^2 or sp^3 hybrid orbital. In the last case, sp^3, three p orbitals mix with a single s orbital, allowing for a total of four bonds. In the case of the sp^3d hybrid orbital, three p orbitals mix with a s and a d orbital allowing a total of five bonds. Let us analyze the formation of hybrid orbitals for the case of methane, CH_4. Carbon has four valence electrons located in 2s and 2p orbitals, each hydrogen has one valence electron located in a 1s orbital. When the C and H combine, the carbon atom transforms into a sp^3 hybrid, leading to four bonds. You can obtain the orbital hybridization in each atom by means of the ABE code. For example, an AB_2E_3 molecule will have five electron regions–resulting of adding the B's and the E's. Table 2 provides the equivalency between the ABE code and the orbital hybridization.

Another example would be BH_3. The chemical bond in this molecule results from the hybridization of boron s orbital with the two p orbital giving a sp^2 hybridization. As the class of the molecule is AB_3, it has three electron regions. Indeed,
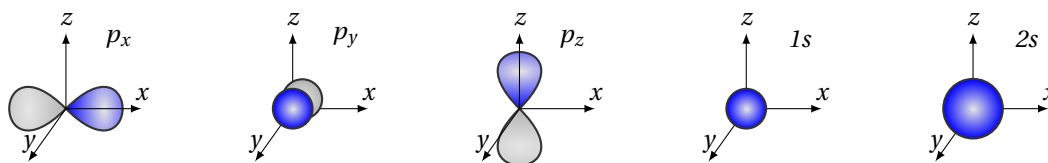
Figure 17: Atomic orbitals of an oxygen atom. From left to right: p_x, p_y, p_z, 1s and 2s. (Bottom left) Hybrid sp^3 orbitals in CH$_4$ resulting of the combination of three p orbitals and the s orbital of C and bonding the s orbitals of hydrogen. (Bottom right) Hybrid sp^2 orbitals in BH$_3$.

according to Table 2, an AB$_3$ has sp^2 hybridization.

Table 5 Equivalency between the ABE code and the orbital hybridization				
ABE Code	Electron Regions	Hybrid	Shape	Bond Angle
AB$_2$, ABE	2	sp		180°
AB$_3$, AB$_2$E,ABE$_3$	3	sp^2		120°
AB$_4$, AB$_3$E,AB$_2$E$_2$,ABE$_3$	4	sp^3		109°
AB$_5$, AB$_4$E,AB$_3$E$_2$,AB$_2$E$_3$	5	sp^{3}d		90° and 120°

Polarity of a molecule

There are polar or non-polar bonds, and the concept of bond polarity is connected to the idea of *electronegativity*. Some atoms in a covalent bond tend to attract more the bond-electrons than the others, and we call this atoms electronegative atoms. An example would be the case of a HF molecule. H is an electropositive atom whereas F is electronegative. This means that in the bond, F will attract more the bond-electrons than H. This different tendency to attract the bond electrons

created a dipole moment in the bond.

$$\overset{\delta+}{\text{H}} \underline{\quad\quad} \overset{\delta-}{\text{F}}$$
$$\longmapsto$$
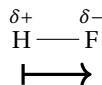
     The electronegativity is a periodic trend that increases from left to right and from bottom to top of the periodic table. Sometimes, these dipole moments in molecules due to the geometry cancel out and the molecule is non-polar as a whole. An example would be CO_2. Every C-O bond is polar, as oxygen is more electronegative than carbon. However, as the molecule is linear the C-O dipole moments cancel out each other and, over all, the molecule is non-polar.

Procedure

Fill the results table and indicate the lewis structure of the molecules–mind some of the molecules do not follow the octet rule. Also indicate the number of electron regions and the hybridization. Finally, identify the geometry of the molecule, the bond angles and the polar character of the molecule–you only need to indicate polar or nonpolar.

> ⚠ CAUTION!
>
>   ⚠ Wear always googles at the lab, even if you do not carry an experiment   **!**

Pre-lab Questions

Molecular Geometry

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1. Compare the electronegativity character of the following pairs of atoms:

   (a) H and F   (b) I and F   (c) H and Cs

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2. Draw the lewis structures of the following molecules: NF_3 and CS_2

3. A molecule results from an sp^3d^2 hybridization. Indicate the geometry of the molecule.

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Results
EXPERIMENT

Molecular Geometry

1. Calculate the number of valence electrons for the following atoms and molecules:

    N _____ $CO_2{}^{2-}$ _____

    O _____ $SO_4{}^{2-}$ _____

    P _____ $ICl_4{}^{-}$ _____

    Br _____ CH_2Cl_2 _____

2. Draw the lewis structure of the following compounds: H_2O, NO, HCl.
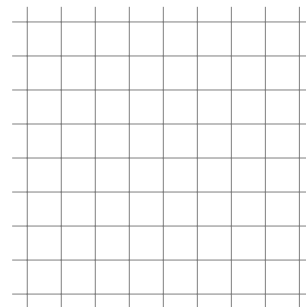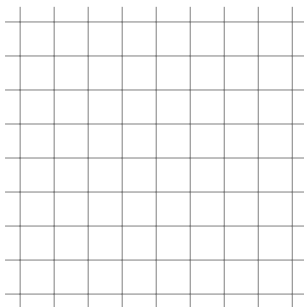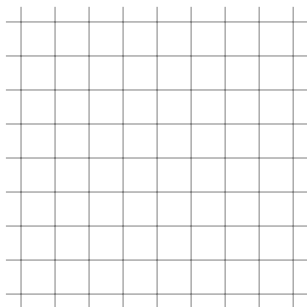
3. Draw the lewis structure of the following compounds and indicate their polarities: CH_4, CH_2Cl_2, $CHCl_3$.
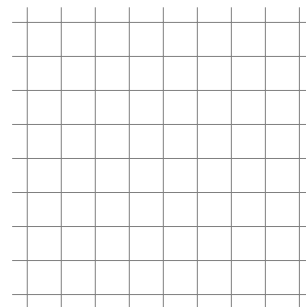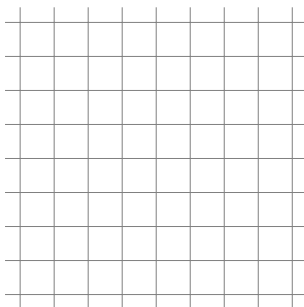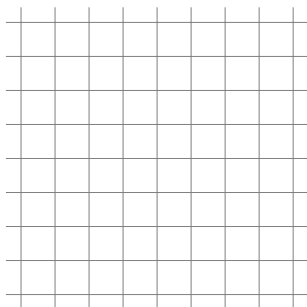
Formula	Lewis Structure	# e^- Regions	Hybridization	Geometry	Angles	Polar?
NH_3	H—N̈—H with H below	4	sp^3	Trigonal Pyramidal	109.5°	Polar
H_2O						
CH_4						
CH_2Cl_2						

Formula	Lewis Structure	# e^- Regions	Hybridization	Geometry	Angles	Polar?
CO_2						
CO_3^{2-}						
CH_2O						
SO_2						

Formula	Lewis Structure	# e^- Regions	Hybridization	Geometry	Angles	Polar?
CH_3OCH_3	C —— O —— C					
ICl_4^- violates octet rule						
C_2H_5OH	C —— C —— O					
C_6H_6	(hexagonal ring of C atoms)					

Formula	Lewis Structure	# e^- Regions	Hybridization	Geometry	Angles	Polar?
$OPCl_3$ violates octet rule						
PCl_5 violates octet rule						
$AlCl_6^{3-}$ violates octet rule						
SO_4^{2-} violates octet rule						

Formula	Lewis Structure	# e^- Regions	Hybridization	Geometry	Angles	Polar?
XeF$_2$ violates octet rule						
SF$_6$ violates octet rule						
BrF$_3$ violates octet rule						
SeF$_4$ violates octet rule						

Periodic Table of Chemical Elements

Daniel Torres

Z	mass
Symbol	
Name	

1 IA	2 IIA	3 IIIB	4 IVB	5 VB	6 VIB	7 VIIB	8 VIIIB	9 VIIIB	10 VIIIB	11 IB	12 IIB	13 IIIA	14 IVA	15 VA	16 VIA	17 VIIA	18 VIIIA
1 1.0079 **H** Hydrogen																	**2** 4.0025 **He** Helium
3 6.941 **Li** Lithium	**4** 9.0122 **Be** Beryllium											**5** 10.811 **B** Boron	**6** 12.011 **C** Carbon	**7** 14.007 **N** Nitrogen	**8** 15.999 **O** Oxygen	**9** 18.998 **F** Flourine	**10** 20.180 **Ne** Neon
11 22.990 **Na** Sodium	**12** 24.305 **Mg** Magnesium											**13** 26.982 **Al** Aluminium	**14** 28.086 **Si** Silicon	**15** 30.974 **P** Phosphorus	**16** 32.065 **S** Sulphur	**17** 35.453 **Cl** Chlorine	**18** 39.948 **Ar** Argon
19 39.098 **K** Potassium	**20** 40.078 **Ca** Calcium	**21** 44.956 **Sc** Scandium	**22** 47.867 **Ti** Titanium	**23** 50.942 **V** Vanadium	**24** 51.996 **Cr** Chromium	**25** 54.938 **Mn** Manganese	**26** 55.845 **Fe** Iron	**27** 58.933 **Co** Cobalt	**28** 58.693 **Ni** Nickel	**29** 63.546 **Cu** Copper	**30** 65.39 **Zn** Zinc	**31** 69.723 **Ga** Gallium	**32** 72.64 **Ge** Germanium	**33** 74.922 **As** Arsenic	**34** 78.96 **Se** Selenium	**35** 79.904 **Br** Bromine	**36** 83.8 **Kr** Krypton
37 85.468 **Rb** Rubidium	**38** 87.62 **Sr** Strontium	**39** 88.906 **Y** Yttrium	**40** 91.224 **Zr** Zirconium	**41** 92.906 **Nb** Niobium	**42** 95.94 **Mo** Molybdenum	**43** 96 **Tc** Technetium	**44** 101.07 **Ru** Ruthenium	**45** 102.91 **Rh** Rhodium	**46** 106.42 **Pd** Palladium	**47** 107.87 **Ag** Silver	**48** 112.41 **Cd** Cadmium	**49** 114.82 **In** Indium	**50** 118.71 **Sn** Tin	**51** 121.76 **Sb** Antimony	**52** 127.6 **Te** Tellurium	**53** 126.9 **I** Iodine	**54** 131.29 **Xe** Xenon
55 132.91 **Cs** Caesium	**56** 137.33 **Ba** Barium	57-71 **La-Lu**	**72** 178.49 **Hf** Halfnium	**73** 180.95 **Ta** Tantalum	**74** 183.84 **W** Tungsten	**75** 186.21 **Re** Rhenium	**76** 190.23 **Os** Osmium	**77** 192.22 **Ir** Iridium	**78** 195.08 **Pt** Platinum	**79** 196.97 **Au** Gold	**80** 200.59 **Hg** Mercury	**81** 204.38 **Tl** Thallium	**82** 207.2 **Pb** Lead	**83** 208.98 **Bi** Bismuth	**84** 209 **Po** Polonium	**85** 210 **At** Astatine	**86** 222 **Rn** Radon
87 223 **Fr** Francium	**88** 226 **Ra** Radium	89-103 **Ac-Lr** Actinide	**104** 261 **Rf** Rutherfordium	**105** 262 **Db** Dubnium	**106** 266 **Sg** Seaborgium	**107** 264 **Bh** Bohrium	**108** 277 **Hs** Hassium	**109** 268 **Mt** Meitnerium	**110** 281 **Ds** Darmstadtium	**111** 280 **Rg** Roentgenium	**112** 285 **Uub** Ununbium	**113** 284 **Uut** Ununtrium	**114** 289 **Uuq** Ununquadium	**115** 288 **Uup** Ununpentium	**116** 293 **Uuh** Ununhexium	**117** 292 **Uus** Ununseptium	**118** 294 **Uuo** Ununoctium

57 138.91 **La** Lanthanum	58 140.12 **Ce** Cerium	59 140.91 **Pr** Praseodymium	60 144.24 **Nd** Neodymium	61 145 **Pm** Promethium	62 150.36 **Sm** Samarium	63 151.96 **Eu** Europium	64 157.25 **Gd** Gadolinium	65 158.93 **Tb** Terbium	66 162.50 **Dy** Dysprosium	67 164.93 **Ho** Holmium	68 167.26 **Er** Erbium	69 168.93 **Tm** Thulium	70 173.04 **Yb** Ytterbium	71 174.97 **Lu** Lutetium
89 227 **Ac** Actinium	90 232.04 **Th** Thorium	91 231.04 **Pa** Protactinium	92 238.03 **U** Uranium	93 237 **Np** Neptunium	94 244 **Pu** Plutonium	95 243 **Am** Americium	96 247 **Cm** Curium	97 247 **Bk** Berkelium	98 251 **Cf** Californium	99 252 **Es** Einsteinium	100 257 **Fm** Fermium	101 258 **Md** Mendelevium	102 259 **No** Nobelium	103 262 **Lr** Lawrencium

- Alkali Metal
- Alkaline Earth Metal
- Metal
- Metalloid
- Non-metal
- Halogen
- Noble Gas
- Lanthanide/Actinide