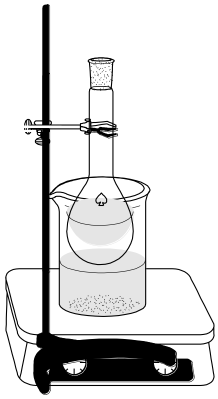
Chemistry M01B
Laboratory Manual



Compiled by the Department of Chemistry at Moorpark College

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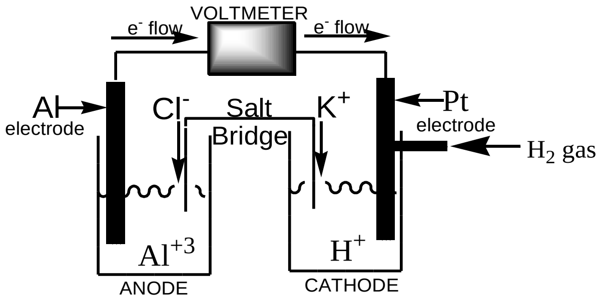
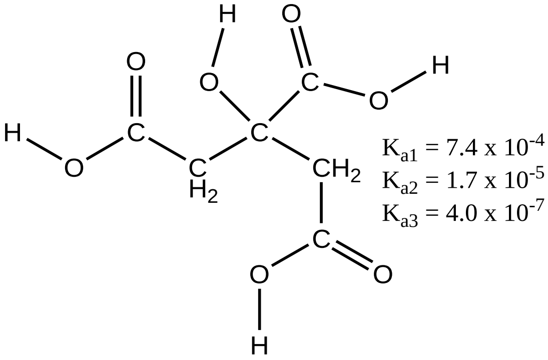


$Al \mid Al^{3+} \parallel H^+ \mid H_2 \mid Pt$

Citric acid ($H_3C_6H_5O_7$)



$K_{a1} = 7.4 \times 10^{-4}$
$K_{a2} = 1.7 \times 10^{-5}$
$K_{a3} = 4.0 \times 10^{-7}$

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Experiment #1 – Calorimetry: Heat of Solution

When a substance undergoes a change in temperature, the quantity (Q) of heat lost or gained can be calculated using the mass (m), specific heat (s), and change in temperature ($\Delta T = T_{final} - T_{initial}$):

$$Q\text{ (heat)} = m\ s\ \Delta T \qquad (1)$$

The specific heat (s) is the amount of heat required to change the temperature of one gram of a particular substance by one degree Celsius (or 1 K). For water, the specific heat is 1.00 calorie / g °C or 4.184 Joules / g °C.

Heat exchange experiments are conducted in a calorimeter which is an insulated container. The calorimeter is constructed such that ideally there will be no heat exchange between the contents of the calorimeter and the surrounding environment. However, heat exchanges can occur between the components within the calorimeter. This can be expressed in the following way:

$$Q_{system} = Q_{gain} + Q_{loss} \qquad (2)$$

If the system does not exchange heat with the surroundings, then the $Q_{system} = 0$, and

$$Q_{gain} = -Q_{loss} \qquad (3)$$

When a hot substance comes in contact with a cold substance within a calorimeter, heat will spontaneously pass from the hot to the cold substance until they achieve a thermal equilibrium (the same constant temperature). The quantity of heat lost and gained is equal in magnitude, but opposite in sign. For example, if a piece of hot metal is placed in cold water within a calorimeter, the following thermodynamic equation can be used:

$$Q_{water} = -Q_{metal} \qquad (4)$$

Since each substance undergoes a temperature change, we can write $Q_{water} = (m\ s\ \Delta T)_{water}$ and $Q_{metal} = (m\ s\ \Delta T)_{metal}$. Substituting this in the above relationship, 4, we get:

$$(m\ s\ \Delta T)_{water} = -(m\ s\ \Delta T)_{metal} \qquad (5)$$

When a chemical reaction occurs in a water solution, the situation is similar to when a hot piece of metal is placed in water. When a reaction occurs, there is a change in the potential energy of the chemicals, resulting in a heat exchange in the calorimeter. As in the hot metal experiment, the heat flow of the reaction is equal in magnitude but opposite in sign to that of the solution as shown by the following equation:

$$Q_{reaction} = -Q_{solution} = -(m\ s\ \Delta T)_{solution} \qquad (6)$$

By measuring the mass of the solution (the water plus the solute) and by observing the temperature change that the solution undergoes, we can calculate $Q_{solution}$ and therefore $Q_{reaction}$. Because the experiment is performed under constant pressure conditions, the heat flow of the reaction is also equal to the enthalpy change, ΔH, for the reaction. Since the mass of solute can vary for different experiments, the $Q_{reaction}$ will also vary. Therefore, ΔH is generally reported as the heat of reaction per a fixed amount of the reacting chemical, and we will calculate ΔH per gram or per mole of the solute.

$$\Delta H_{rxn} = \frac{Q_{rxn}}{mass_{solute}} \quad \text{or} \quad \Delta H_{rxn} = \frac{Q_{rxn}}{mole_{solute}} \qquad (7)$$

If the temperature of the water goes up, heat has been given off by the reaction, so that reaction is exothermic, and ΔH of the reaction is negative. If the temperature of the water goes down, heat is absorbed by the reaction, therefore the reaction is endothermic, and ΔH for the reaction is positive.

Procedure

Construct a calorimeter, where two polystyrene coffee cups with a plastic cover on top are nested together. Weigh and record the mass of the clean, dry calorimeter. Place the calorimeter into a 400 mL glass beaker for stability during the experiment.

Place about 50 mL of distilled water in the calorimeter and weigh. Measure the temperature of the water to 0.01°C. The temperature should be within a degree or two of room temperature. Weigh out about 5 grams of the solid assigned to you. Add the solid to the calorimeter. Stirring and swirling the calorimeter, determine to 0.01°C the stable maximum or minimum temperature reached as the solid dissolves. Check to make sure that the solid is completely dissolved. A temperature change of at least five degrees should be obtained in this experiment. AFTER THE FINAL TEMPERATURE IS DETERMINED, WEIGH THE CALORIMETER WITH THE DISSOLVED SOLID.

Repeat the experiment for trial 2. If necessary, increase the amount of solid used to obtain a temperature change of at least five degrees. Use the same calorimeter containing a new 50 mL sample of water. Assume the same mass for the dry calorimeter.

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Data and Calculations

	Trial 1		Trial 2	
Mass of empty calorimeter	_______	=	_______	g
Mass of calorimeter + water	_______		_______	g
Mass of calorimeter + water + solid (final mass)	_______		_______	g
Mass of solution	_______		_______	g
Mass of solid (solute)	_______		_______	g
Initial temperature	_______		_______	°C
Final temperature	_______		_______	°C
ΔT	_______		_______	°C
$Q_{solution} = (m\ s\ \Delta T)_{solution}$	_______		_______	J
$Q_{rxn} = -Q_{solution}$	_______		_______	J

Calculate the heat of reaction, ΔH, per gram of solid

$$\Delta H_{reaction} = \frac{Q_{rxn}}{mass_{solute}}$$

___________J / g (trial 1)* and ___________J / g (trial 2)*

* *Note*: These values should be within 5% of each other or another trial should be done.

Average ΔH per gram _____________ J / g

Molecular Formula of Solid __________________ Molecular Weight _____________

Calculate the molar heat of solution, ΔH, for this solid. Use the average ΔH value.

$\Delta H_{reaction}$ ______________ kJ / mol

Calculate the % error for the above value.
(Literature values: Na_2CO_3 $\Delta H_{reaction} = -28.1$ kJ / mol; NH_4NO_3 $\Delta H_{reaction} = +25.7$ kJ / mol)

Theoretical $\Delta H_{reaction}$ _________ kJ / mol % error _________

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Post-Lab Questions: Heat of Solution

1. A metal sample weighing 63.2 g with a temperature of 100.0 °C was placed in a calorimeter containing 41.0 g of water at 24.5 °C. The equilibrium temperature of the water and metal was found to be 35.0 °C.

   A. What was ΔT for the water? ($\Delta T = T_{final} - T_{initial}$) __________ °C

   B. What was ΔT for the metal? __________ °C

   C. Taking the specific heat of water to be 4.184 J / g °C, calculate the specific heat of the metal using Eq. 5.

   __________ J / g °C

2. When 5.00 g of KNO_3 were dissolved in 49.00 g H_2O at 24.00 °C inside a calorimeter, the temperature of the resulting solution fell to 15.60 °C.

   A. Is this reaction endothermic or exothermic? __________________
   Explain:

   B. Calculate the heat lost or gained by the solution chemicals (this is Q_{soln}) in the calorimeter.

   Q_{soln} = _________ Joules

   C. What is Q_{rxn} for the reaction that occurred?

   Q_{rxn} = _________ Joules

   D. Calculate the heat of this reaction, ΔH, in Joules/g KNO_3.

   ΔH_{rxn} = _________ Joules / g

   E. Calculate the molar heat of this reaction, ΔH, in kJ/mol KNO_3.

   ΔH_{rxn} = _________kJ / mole

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Experiment #2 – Hess's Law: Heat of Formation of MgO

Hess's Law states that when a reaction is carried out in a series of steps, ΔH for the overall reaction will equal the sum of the enthalpy changes in the individual steps.
It is possible to calculate enthalpy of a reaction in both the lab setting, and by using Appendix values. Enthalpy is an extensive property, so the amount is important. If a series of reactions are used to create an overall reaction, the enthalpy values can be added to solve for the overall enthalpy of a reaction.

$$\Delta H_{rxn} = \Delta H_a + \Delta H_b + \Delta H_c \qquad (1)$$

In this experiment, the enthalpy of each reaction step will be measured using a calorimeter. The heat released by the reaction will be absorbed by the calorimeter such that:

$$\Delta H_{rxn} = \frac{-Q_{cal}}{\text{moles of solid}} \qquad (2)$$

The heat absorbed by the calorimeter (Q_{cal}) can be found using the equation:

$$Q_{cal} = ms\Delta T \qquad (3)$$

As an alternative to a lab determination of enthalpy values, any reaction may be broken down to formation reactions from elements. Appendix values may be looked up to solve for the enthalpy of reaction under standard conditions.

$$\Delta H_{rxn}° = \Sigma\ nH_f° \text{ (products)} – nH_f° \text{ (reactants)} \qquad (4)$$

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Part A Procedure

Assemble a calorimeter consisting of two polystyrene coffee cups nested together, weigh and record the mass of the dry calorimeter, and place in a 400 mL beaker to stabilize. Obtain a 10 cm piece of Mg, then clean, weigh, and record its mass. Place about 50 mL of 3.0 M HCl in the calorimeter and determine its temperature to the nearest 0.01 °C. Add the Mg metal to the HCl solution and quickly cover the calorimeter. While constantly swirling the solution, determine and record the maximum temperature (0.01 °C) achieved. When complete, weigh and record the mass of the calorimeter and final solution. Calculate ΔH per mole of Mg for the reaction. *Repeat the experiment until two ΔH values agree to within 5% of each other.*

Part B Procedure

Use the same calorimeter from Part A and its original empty mass. Pour about 50 mL of 6.0 M HCl in the calorimeter, measure, and record its temperature to the nearest 0.01 °C. Place approximately 1 gram of MgO in a weighing dish and record its exact mass. Add the MgO to the HCl solution in the calorimeter and follow the procedure from Part A to determine the final temperature of the solution. After the MgO is added to the calorimeter, obtain the mass of the empty weighing dish in order to determine the amount of MgO added to the calorimeter. Weigh and record the mass of the calorimeter and final solution. Calculate ΔH per mole of MgO for the reaction. *Repeat the experiment until two ΔH values fall within 5% of each other*.

Note*: Specific heat of the hydrochloric acid solution in both Part A and Part B is 3.70 J / g °C. *This is not water.

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Data for Part A: Clearly show calculations on another space, (s) = 3.70 J / g °C

Mass of Mg ________________ ________________

Mass of Empty Calorimeter + Lid ________________ = ________________

Mass of Calorimeter + Lid + Final Solution ________________ ________________

Initial Temperature of HCl Solution ________________ ________________

Final Temperature of Solution ________________ ________________

Mass of Final Solution ________________ ________________

ΔT of Solution ________________ ________________

ΔH for reaction **Part A**, kJ/mole Mg ________________ ________________

Average ΔH_A ______________ kJ / mol Mg

Data for Part B: Clearly show calculations on another space, (s) = 3.70 J / g °C

Mass of Empty Calorimeter + Lid ________________ = ________________

Mass of Calorimeter + Lid + Final Solution ________________ ________________

Mass of Weighing Dish ________________ ________________

Mass of Weighing Dish + MgO ________________ ________________

Initial Temperature of HCl ________________ ________________

Final Temperature of Solution ________________ ________________

ΔT of Solution ________________ ________________

Mass of MgO ________________ ________________

Mass of Final Solution ________________ ________________

ΔH for reaction **Part B**, kJ / mole MgO ________________ ________________

Average ΔH_B ______________ kJ / mol MgO

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Data Analysis and Calculations

Use Hess's Law and the following information to determine the heat of formation of MgO:

A. $Mg(s) + 2\ HCl\ (aq) \rightarrow H_2(g) + MgCl_2(aq)$ ΔH_A = ____________
(**Part A**)

B. $MgO(s) + 2\ HCl\ (aq) \rightarrow H_2O(l) + MgCl_2(aq)$ ΔH_B = ____________
(**Part B**)

C. Write the formation equation for liquid water

$\Delta H_f°$ = ____________
Use appendix for value

D. Write the formation equation for MgO(s)

E. Use Hess's Law and the above information to calculate the heat of formation for MgO(s). Show all your work.

F. Look up the literature value of $\Delta H_f°$ for MgO and calculate the % error.

Theoretical $\Delta H_f°$ MgO ______________ kJ / mol % error ___________

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Experiment #3 – Bomb Calorimeter: Heat of Combustion of 1-Butanol

Calorimetry can be used to study the chemical potential energy stored in substances. One of the most important types of reactions studied using calorimetry is combustion, in which a compound reacts completely with excess oxygen. In particular, this lab involves measuring the heat accompanying the combustion of 1-butanol (C_4H_9OH) using a bomb calorimeter at constant volume.

This organic alcohol is placed in a small cup within a sealed vessel called a *bomb*. The bomb, which is designed to withstand high pressures, has an inlet valve for adding oxygen and electrical contacts to initiate the combustion. After the sample has been placed in the bomb, the bomb is sealed and pressurized with oxygen. It is then placed in the calorimeter and covered with an accurately measured quantity of water. When all the components within the calorimeter have come to the same temperature, the combustion reaction is initiated by passing an electrical current through a fine wire that is just above the sample. When the wire gets sufficiently hot, the sample ignites.

Procedure

1. Clean and dry all equipment: bucket, bomb and sample cup.

2. Add 2000 mL of D.I. water to the metal bucket and place it in the calorimeter paying attention to the guides.

3. Attach a 10 cm length of fuse wire to the sample holder terminals. Place the wire through the holes and wrap it once or twice around each terminal.

4. Place the sample cup in the sample holder and adjust the fuse wire loop so that it hangs approximately halfway into the cup.

5. Pipet 1.00 mL of D.I. water into the bottom of the bomb chamber (not into sample cup).

6. Pipet 1.00 mL of 1-butanol into the cup; make sure to perform this after step 5 so that the sample does not evaporate.

7. Place the cup containing the 1-butanol into the bomb and secure the cover a gentle hand-tight.

8. Pressurize the bomb with approximately 20 atm of oxygen gas. <u>Your instructor MUST be present during this portion of the procedure</u>.

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9. Place the bomb in the calorimeter, attach the ignition wires to the top of bomb, cover the calorimeter by aligning the pilot hole and screw, and turn on the stirrer.

10. Wait 5 minutes and observe the temperature at 30 second intervals until a constant equilibrium temperature is achieved (4 constant values). Record this temperature.

11. Plug in the wires onto the ignition unit and press the button to ignite your sample.

12. Continue to observe the temperature until a constant equilibrium temperature is achieved. Record this temperature.

13. Carefully remove the cover and place it in the holder.

14. Remove the bomb, equalize the pressure, disassemble and completely dry all parts of the bomb.

15. If successive trials are to be run, add a new 2000 mL supply of D.I. water to the bucket and repeat steps 3–14.

Data and Calculations

Volume of sample _______________ Density of sample _______________

Initial temperature _______________ Final temperature _______________

1. Write a balanced equation for the combustion reaction of one mole of 1-butanol.

2. Calculate the mass (in g) of the 1-butanol sample.

3. Calculate the heat of reaction per mole of 1-butanol. ***Our calorimeter constant (Heat Capacity) = 10.3 kJ / °C***

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4. Does question #3 solve for ΔE (internal energy) or ΔH (enthalpy)? Circle your choice and briefly explain.

5. Calculate the **ideal** work per mole of alcohol for the combustion reaction at 1 atm and 25 °C.

6. Give the following values in kJ/mol for $CH_3(CH_2)_2CH_2OH$ at 1 atm and 25°C:
   Show your work below for full credit.

   ΔE = _______________ ΔH = _______________ w = ________________

7. Use literature values of enthalpy of formation to calculate ΔH_{rxn} for the balanced combustion reaction in question #1. Use this as the theoretical value.

8. Calculate a % error for this experiment using ΔH_{rxn} values from problems 6 and 7 above.

9. Why is 1 mL of D.I. water added to the bottom of the bomb chamber?

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Experiment #4 – Chemical Kinetics: The Iodination of Acetone

The rate at which a chemical reaction occurs depends on several factors: the nature of the reaction, the concentrations of the reactants, the temperature, and the presence of possible catalysts. All of these factors can markedly influence the observed rate of reaction.

In this experiment, we will study a reaction which, in the vicinity of room temperature, proceeds at a relatively easily measured rate.

For a given reaction, the rate typically increases with an increase in the concentration of any reactant. For the reaction $aA + bB \rightarrow cC$, the rate can be expressed by the following equation, which is called the rate law,

$$\text{rate} = k[A]^m[B]^n \qquad (1)$$

where m and n are generally, but not always, integers, 0, 1, 2 or possibly 3; [A] and [B] are the initial concentrations of A and B (ordinarily in moles per liter); and k is the specific rate constant for the reaction. The numbers m and n are called the orders of the reaction with respect to A and B. If m is 1 the reaction is said to be first order with respect to the reactant A. If n is 2 the reaction is second order with respect to reactant B. The overall order is the sum of m and n. In this example, the reaction would be third order overall.

The rate of reaction is also significantly dependent on the temperature at which the reaction occurs. An increase in temperature increases the rate, an often cited rule being that a 10 ^{o}C rise in temperature will double the rate. This rule is only approximately correct; nevertheless, it is clear that a rise of temperature to say 100 ^{o}C could change the rate of a reaction appreciably.

As with the concentration, there is a quantitative relationship between reaction rate and temperature. This relation is based on the idea that in order to react, the reactant species must have a certain minimum amount of energy present at the time the reactants collide in the reaction step. This amount of energy, which is typically furnished by the kinetic energy of the species present, is called the activation energy of the reaction.

The equation relating the rate constant, k, to the absolute temperature, T, and the activation energy, E_a, is

$$\ln k = \frac{-E_a}{RT} + \text{constant} \qquad (2)$$

where R is the gas constant (8.314 Joules/mole K for E_a in Joules per mole). By measuring k at different temperatures we can determine graphically the activation energy for a reaction.

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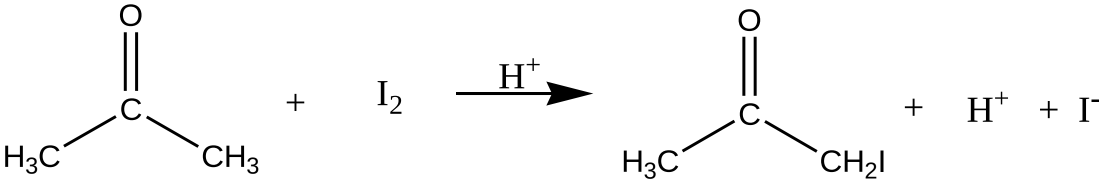
In this experiment, we will study the kinetics of the reaction between iodine and acetone in aqueous solution:



The rate of this reaction is expected to depend on the concentration of hydrogen ion in the solution, a catalyst, as well as the concentrations of the two reactants. By Equation 1, the rate law for this reaction is

$$\text{rate} = k\,[\text{acetone}]^m\,[H^+]^n\,[I_2]^p \qquad (3)$$

where **m**, **n**, and **p** are the orders of the reaction with respect to **acetone**, **hydrogen ion**, and **iodine**, respectively, and k is the rate constant for the reaction.

The rate of this reaction can be expressed as the (small) change in the concentration of I_2, $\Delta[I_2]$, divided by the time interval, Δt, required for the change:

$$\text{rate} = \frac{-\Delta[I_2]}{\Delta t} \qquad (4)$$

The minus sign is to make the rate positive ($\Delta[I_2]$ is negative). Ordinarily, since rate varies as the concentrations of the reactants according to Equation 3, in a rate study it would be necessary to measure (directly or indirectly) the concentration of each reactant as a function of time; the rate would typically vary markedly with time, decreasing to very low values as the concentration of at least one reactant becomes very low. This makes reaction rate studies relatively difficult to carry out and introduces mathematical complexities that are difficult for beginning students to understand.

The iodination of acetone is a rather atypical reaction in that it can be very easily investigated experimentally. First of all, iodine has color so that one can readily follow changes in iodine concentration visually. A second and very important characteristic of this reaction is that it turns out to be zero-order in I_2 concentration. This means (see Equation 3) that the rate of the reaction does not depend on $[I_2]$ at all as $[I_2]^0 = 1$ no matter what the value of $[I_2]$ is as long as it is NOT zero.

Since the rate of reaction does not depend on $[I_2]$, we can study the rate by simply making I_2 the limiting reagent present in a large excess of acetone and H^+ ion. We then measure the time required for a known initial concentration of I_2 to be completely used up. If both acetone and H^+ are present at much higher concentrations than that of I_2, their concentrations will not change appreciably during the course of the reaction, and the rate will remain, by Equation 3, effectively constant until all the iodine is gone, at which time the reaction will stop. Under such circumstances, if it takes t seconds for the color of a solution having an initial concentration of I_2 equal to $[I_2]_{\text{initial}}$ to disappear, the rate of reaction, by Equation 4, would be:

$$\text{rate} = \frac{-\Delta[I_2]}{\Delta t} = \frac{[I_2]_{\text{initial}} - 0}{t - 0} = \frac{[I_2]_{\text{initial}}}{t} \qquad (5)$$

Although the rate of the reaction is constant during its course under the conditions we have set up, we can vary it by changing the initial concentrations of acetone and H^+ ion. If, for example, we should double the initial concentration of acetone over that in Mixture 1, keeping $[H^+]$ and $[I_2]$ at the same values they had previously, then the rate of Mixture 2 would, according to Equation 3, be different from that in Mixture 1:

$$\text{rate 2} = k\,[2A]^m\,[H^+]^n\,[I_2]^p \qquad (6a)$$
$$\text{rate 1} = k\,[A]^m\,[H^+]^n\,[I_2]^p \qquad (6b)$$

Dividing the first equation by the second, we see that the k's cancel, as do the terms in the iodine and hydrogen ion concentration, since they have the same values in both reactions, and we obtain simply

$$\frac{\text{rate 2}}{\text{rate 1}} = \frac{[2A]^m}{A^m} = 2^m \qquad (6)$$

Having measured both rate 2 and rate 1 by Equation 5, we can find their ratio, which must be equal to 2^m. We can then solve for m using logarithms and so find the order of the reaction with respect to acetone:

$$\log(\text{rate 2/rate 1}) = \log 2^m = m \log 2 \qquad (7)$$

By a similar procedure we can measure the order of the reaction with respect to H^+ ion concentration and also confirm the fact that the reaction is zero-order with respect to I_2. Having found the order with respect to each reactant, we can then evaluate k, the rate constant for the reaction.

The experiment goals include the determination of the orders m and n, the confirmation of the fact that p, the order with respect to I_2, equals zero, and the evaluation of the rate constant k for the reaction at room temperature. You will be furnished with standard solutions of acetone, iodine, and hydrogen ion, and with the composition of the solutions that will give a reasonable rate.

The final part of the experiment is to study the rate of this reaction at different temperatures in order to find its activation energy. This is done by studying the rate of reaction in one of the mixtures at room temperature and at two other temperatures, one above and one below room temperature. Knowing the rates, and hence the k's, at the three temperatures, you can solve for E_a, the energy of activation, by plotting ln k vs. 1/T. The slope of the resultant straight line, by Equation 2, is $-E_a/R$.

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Procedure

Select two regular test tubes; when filled with distilled water, they should appear to have identical color when you view them down the tubes against a white background. One test tube will be the reaction test tube and the other will be the reference test tube. For the reference, add 5 mL of D.I. water to a test tube and set aside for comparison later.

For each trial, add the specified volumes of 1.0 M HCl, 4.0 M acetone, and water from the provided burets directly into the clean, dry reaction test tube. Swirl the test tube to mix the reagents.

Noting the time, add the iodine solution into the test tube using the appropriate pipet and quickly swirl the test tube or stir with a CLEAN, DRY STIRRING ROD to thoroughly mix the reagents. The reaction mixture will appear yellow because of the presence of the iodine, and the color will fade slowly as the iodine reacts with the acetone. Look down the reacting test tube side-by-side with the reference test tube toward a well-lit piece of white paper, and note the time the color of the iodine just disappears. Measure the temperature of the mixture in the test tube after the reaction is complete.

Repeat the experiment using the reacted solution as the reference instead of distilled water. The amount of time required in the two runs should agree within 20 seconds. Use this same reference test tube for all the remaining mixtures.

Carry out the remaining reaction mixtures twice with each of the indicated compositions (including your own mixture composition); the times should not differ by more than 15 seconds for the same mixture. The temperature must remain relatively constant, within two degrees of that in the initial run.

Calculate the new initial (diluted) concentrations for the reactants ($M_1V_1 = M_2V_2$) and the rates of the reaction. The rate of the reaction equals the initial concentration of I_2 in the reaction mixture divided by the average elapsed time. Since the reaction is zero-order in I_2, and since both acetone and H^+ ion are present in great excess, the rate is constant throughout the reaction and the concentrations of both acetone and H^+ remain essentially at their initial values in the reaction mixture.

Calculate the order with respect to each reactant for this reaction. Then determine the value of the rate constant, k, from the rate law (3) using your reactant orders, the calculated concentrations, and rate data for each of the mixtures you studied. If the temperatures at which the reactions were run are all within two degrees, k should be about the same for each mixture.

Using your own mixture values, predict how long it will take for the I_2 color to disappear. Calculate the predicted reaction rate using the rate law with your average k value, your calculated orders, and the initial concentrations for your mixture. Then calculate the predicted time using equation 5. Compare your measured time for this reaction with your predicted time.

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Reaction data from mixture D at three temperatures has been provided for you to determine the energy of activation for the reaction using Equation 2.

Data and Calculations

Table 1. Reactant Volumes and Reaction Times

Mixture	Volume (mL) of:				Time of reaction (sec)			Temp
	4.0 M acetone	1.0 M HCl	0.0050 M I_2	H_2O	1st run	2nd run	Ave time	°C
A	1.00	1.00	1.00	2.00				
B	2.00	1.00	1.00	1.00				
C	1.00	2.00	1.00	1.00				
D	1.00	1.00	0.50	2.50				
Your Mixture*								

*Note: Must add up to 5.0 mL; water is not required.

Table 2. Initial Concentration and Rate of Reaction

Mixture	$[\text{acetone}]_{\text{ini}}$	$[H^+]_{\text{ini}}$	$[I_2]_{\text{ini}}$	Rate = $[I_2]_{\text{ini}}$ / (ave time)
A				
B				
C				
D				
Your Mixture*				

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Part A: Reactant Order Determination

Calculate the value of m (order with respect to acetone) to TWO decimal places. This means you have to use logarithms. Show calculations. Then round off to the nearest integer to get the true value for m.

m = _________ (2 decimal places) m = _________ (nearest integer)

Calculate the value of n (order with respect to the H^+):

n = _________ (2 decimal places) n = _________ (nearest integer)

Calculate the value of p (order with respect to the I_2):

p = _________ (2 decimal places) p = _________ (nearest integer)

Write the overall rate law: ____

Part B: Determination of the Rate Constant, k

Given the values of m, n, and p, calculate the rate constant k (**with correct units**) for each mixture by simply substituting those orders, the initial concentrations, and the observed rate from the Table 2 above into Equation 3.

Table 3. Rate Constant, k

Mixture	A	B	C	D	average	Units of k
k						

Show calculations for k value of Mixture A:

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Part C: Prediction of Reaction Rate in Your Mixture

Initial concentrations (from Table 2):

$[\text{acetone}]_{\text{ini}}$ __________ M $[H^+]_{\text{ini}}$ __________ M $[I_2]_{\text{ini}}$ __________ M

Calculate the predicted rate based on your determined rate law in Part A as well as your average rate constant (from Part B).

Predicted rate: ________________ M/sec

Calculate the predicted ***time*** (in seconds) for reaction based on $[I_2]_{\text{ini}}$ and the value for the predicted rate above using:

Rate = $[I_2]_{\text{ini}}$ / (time)

Predicted time: __________ sec

Observed average time for reaction ________ sec (from Table 1 above)

Calculate the percent difference between the observed and predicted times below:

% difference: ______________

Name: ___________________________________ Section: ______________________

Part D: Energy of Activation Determination

PROBLEM SET – Use provided data below

Fill Table 4 below.

Table 4. Reaction Rate Data for Mixture **D**.

Temp (°C)	Temp (Kelvin)	Time (sec)	Rate = $[I_2]_{ini}$ / (time) ***(NOTE:** Use $[I_2]_{ini}$ from Mixture D)*
10.		208	
22		93	
40.		28	

Fill Table 5 below by calculating the rate constant at each temperature using rate from Table 4 above, initial concentrations for Mixture D (Table 2) and your experimentally determined rate law (from Part A).

Table 5. Calculated Values for Arrhenius Plot

Temperature	k	ln k *(y-axis)* (two decimal places)	1 / T *(x-axis)* (Kelvin^{-1})
10 °C			
22 °C			
40 °C			

Construct an Arrhenius plot by graphing ln k vs. 1 / T. Find the slope of the best fitting (straight) line through the points. Show your calculations below: Alternatively, you can use MS Excel to construct your graph; write the trendline equation below.

Slope = ___________ OR Trendline Equation (MS Excel): ______________________

From Equation 2, we see that the slope = $-E_a / R$ where R = 3.814 J / mol K. Use this relationship to calculate the value of E_a:

E_a = ___________________ kJ / mol

Name: ____________________________________ Section: ______________________

Pre-Lab Questions: The Iodination of Acetone

1. In a reaction involving the iodination of acetone, the following volumes were used to make up the reaction mixture.

   1.0 mL 4.0 M acetone + 1.0 mL 1.0 M HCl + 1.0 mL 0.0050 M I_2 + 2.0 mL H_2O

   a. Calculate the initial (diluted) concentration of acetone in the reaction mixture.

   $[\text{acetone}]_{\text{ini}}$ = ___________

   b. Calculate the initial (diluted) concentration of the hydrogen ion, H^+, in the reaction mixture.

   $[H^+]_{\text{ini}}$ = ___________

   c. Calculate the initial (diluted) concentration of iodine, I_2, in the reaction mixture.

   $[I_2]_{\text{ini}}$ = ___________

2. Using the reaction mixture in Problem 1, a student found that it took 300 seconds for the color of the I_2 to disappear.

   a. What was the rate of the reaction?

   rate = ________________

   b. Given the rate from Part a and the initial concentrations of acetone, H^+ ion, and I_2 in the reaction mixture, write Equation 3 as it would apply to the mixture.

   rate =

   c. What are the unknowns that remain in the equation in Part b?

   ________________ ________________ ________________ ________________

Name: ________________________________ Section: ____________________

3. A second reaction mixture was made up in the following way:

   2.0 mL 4.0 M acetone + 1.0 mL 1.0 M HCl + 1.0 mL 0.0050 M I_2 + 1.0 mL H_2O

   a. What were the initial concentrations of acetone, H^+ ion, and I_2 in the reaction mixture?

      $[\text{acetone}]_{\text{ini}}$ ___________ M; $[H^+]_{\text{ini}}$ ___________ M; $[I_2]_{\text{ini}}$ ___________ M

   b. It took 140 seconds for the I_2 color to disappear from the reaction mixture when it occurred at the same temperature as the reaction in Problem 2.

      What was the rate of the reaction? ____________

      Write Equation 3 as it would apply to the second reaction mixture:

      rate =

   c. Solve for the value of m, the order of the reaction with respect to acetone. (Use the logarithm method and calculate the value of m to two decimal places and then round to the nearest integer.)

      m = ________ (2 decimal places) m = ________ (nearest integer)

Name: ________________________________ Section: ____________________

Experiment #5 – Chemical Kinetics: Iodine Clock Reaction

In the previous experiment, we discussed the factors that influence the rate of a chemical reaction and presented the terminology used in quantitative relations in studies of the kinetics of chemical reactions. That material is also pertinent to this experiment and should be studied before you proceed further.

This experiment involves the study of the rate properties, or chemical kinetics, of the following reaction between iodide ion and bromate ion under acidic conditions:

$$6\ I^{-}(aq) + BrO_3^{-}(aq) + 6\ H^{+}(aq) \rightarrow 3\ I_2(aq) + Br^{-}(aq) + 3\ H_2O(l) \qquad (1)$$

This reaction proceeds at an easily measurable rate that depends on the concentrations of the I^-, BrO_3^-, and H^+ ions according to the rate law discussed in the previous experiment. For this reaction, the rate law takes the form

$$\text{rate} = k\,[I^-]^m\,[BrO_3^-]^n\,[H^+]^p \qquad (2)$$

The main goals of the experiment will be to evaluate the reaction orders m, n and p, the rate constant, k, and investigate the manner in which the reaction rate depends on temperature to solve for its activation energy, E_a, for the reaction.

Our method for measuring the rate of the reaction involves what is frequently called a "clock" reaction. In addition to Reaction 1, whose kinetics we will study, the following reaction will also be made to occur simultaneously in the reaction flask:

$$I_2(aq) + 2\ S_2O_3^{2-}(aq) \rightarrow 2\ I^{-}(aq) + S_4O_6^{2-}(aq)$$

As compared with (1) this reaction is essentially instantaneous above 10 °C. The I_2 produced in (1) reacts completely with the thiosulfate, $S_2O_3^{2-}$, ion present in the solution so that once all the thiosulfate ion has reacted, the concentration of I_2 is effectively zero. As soon as the $S_2O_3^{2-}$ is gone from the system, the I_2 produced by (1) remains in the solution, and its concentration begins to increase. The presence of I_2 is made strikingly apparent by a starch indicator which is added to the reaction mixture, since I_2 even in small concentrations reacts with starch solution to produce a blue color.

By carrying out Reaction 1 in the presence of $S_2O_3^{2-}$ and a starch indicator, we introduce a "clock" into the system. Our clock tells us when a given amount of BrO_3^- ion has reacted (1/6 mole BrO_3^- per mole $S_2O_3^{2-}$). The rate of reaction can be expressed in terms of the time it takes for a particular amount of BrO_3^- to be used up. In all our reactions, the amount of $S_2O_3^{2-}$ that reacts will be constant and small, therefore the amount of BrO_3^- to be used up is both constant and small as compared to the initial amounts of reactants. This means that the concentrations of all reactants will be essentially constant in Equation 2, and hence so will the rate during each reaction.

In our experiment, we will carry out the reaction between BrO_3^-, I^-, and H^+ ions under different concentration conditions. Measured amounts of each of these ions in water solution will be mixed in the presence of a constant small amount of $S_2O_3^{2-}$. The time it takes for each mixture to turn blue will be measured. The time obtained for each reaction will be inversely proportional to its rate. By changing the concentration of one reactant and keeping the other concentrations constant, we can investigate how the rate of the reaction varies with the concentration of a particular reactant. Once we know the order for each reactant, we can determine the rate constant for the reaction.

In the last part of the experiment we will investigate how the rate of the reaction depends on temperature. You will recall that in general the rate increases sharply with temperature. By measuring how the rate varies with temperature we can determine the activation energy, E_a, for the reaction by making use of the Arrhenius equation:

$$\ln k = -E_a / RT + \text{constant} \qquad (3)$$

In this equation, k is the rate constant at the Kelvin temperature T, E_a is the activation energy, and R is the gas constant. By plotting ln(k) against 1/T we should obtain, by Equation 3, a straight line whose slope equals $-E_a/R$. From the slope of that line, we can easily calculate the activation energy.

Procedure

Dependence of Reaction Rate on Concentration

In the following table, we have summarized the reagent volumes to be used in carrying out the several reactions whose rates we need to know in order to find the general rate law for Reaction 1.

Reaction Mixtures at Room Temperature (in mL)

Reaction		Reaction Flask I	
Mixture	0.010M KI	0.001 M $Na_2S_2O_3$	H_2O
1	5.0	5.0	5.0
2	10.0	5.0	0
3	5.0	5.0	0
4	5.0	5.0	0
5	4.0	5.0	6.0

Name: ________________________________ Section: ____________________

Reaction Mixture	0.040M $KBrO_3$	Reaction Flask II 0.010 M HCl	starch
1	5.0	5.0	3 or 4 drops
2	5.0	5.0	3 or 4 drops
3	10.0	5.0	3 or 4 drops
4	5.0	10.0	3 or 4 drops
5	2.0	8.0	3 or 4 drops

Since there are several reagents to mix and we don't want the reaction to start until we are ready, we will put some of the reagents into one flask and the rest into another, selecting them so that no reaction occurs until the contents of the two flasks are mixed. For reaction mixture 1, measure out 5.0 mL 0.010 M KI, 5.0 mL 0.001 M $Na_2S_2O_3$, and 5.0 mL distilled water into a 125-mL Erlenmeyer flask (Reaction Flask I). Into a second 125-mL Erlenmeyer flask, measure out 5.0 mL 0.040 M $KBrO_3$, 5.0 mL 0.10 M HCl, and add 3 or 4 drops of starch indicator solution (Reaction Flask II).

Pour the contents of Reaction Flask II into Reaction Flask I and swirl the solutions to mix them thoroughly. Note the time at which the solutions were mixed. Continue swirling the solution. It should turn blue in about 2 minutes. Note and record the time at the instant that a blue color appears. Record the temperature of the blue solution to 0.2 °C.

Repeat the procedure with the other mixtures. *Don't forget to add the starch indicator before mixing the solutions* in the two flasks. The reaction flasks should be rinsed with distilled water and dried between runs. The temperature should remain about the same for all the runs. Repeat any experiments that did not appear to proceed properly.

Dependence of Reaction Rate on Temperature

In this part of the experiment, the reaction will be carried out at several different temperatures using Reaction Mixture 2 in all cases. The temperatures we will use will be room temperature (which you have already done), hot about 40 °C, and cold about 10 °C.

Use your Mixture 2 data for the time and temperature values in the room temperature column of table 2. To determine the time at hot temperature about 40 °C proceed as follows. Make up Reaction Mixture 2 as you did in Part A, including the indicator. However, instead of mixing the solutions in the two flasks at room temperature, place the flasks into hot tap water about 40 °C drawn from the hot water tap into a plastic tub. Check to see that the water is indeed at about 40 °C, and leave the flasks in the water for several minutes to bring them to the proper temperature. Then mix the two solutions, noting the time of mixing. Continue swirling the reaction flask in the warm water. When the color change occurs, note the time and the temperature of the solution in the flask.

Repeat the experiment at about 10 °C, cooling all the reactants in water with a bit of ice added before starting the reaction. Record the time required for the color to change and the final temperature of the reaction mixture.

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Dependence of the Reaction Rate on the Presence of Catalyst

Some ions have a pronounced catalytic effect on the rates of many reactions in water solution. Observe the effect on this reaction by once again making up Reaction Mixture 2. Before mixing, add 1 drop 0.5 M $[NH_4]_2MoO_4$, ammonium molybdate, and a few drops of starch indicator to Reaction Flask II. Swirl the flask to mix the catalyst thoroughly. Then mix the solutions, noting the time required for the color to change. Record this time in the data section.

Data and Calculations

Orders of the Reaction. Rate Constant Determination

For the reaction:

$$6\ I^-(aq) + BrO_3^-(aq) + 6\ H^+(aq) \rightarrow 3\ I_2(aq) + Br^-(aq) + 3\ H_2O(l) \qquad (1)$$

we have the rate law and reaction rate given by:

$$\text{rate} = k\,[I^-]^m\,[BrO_3^-]^n\,[H^+]^p = \frac{-\Delta[BrO_3^-]}{\Delta t} \qquad (4)$$

In all the reaction mixtures used in the experiment, the color change occurred when a constant predetermined number of moles of BrO_3^- had been used up by the reaction. The color "clock" allows you to measure the time required for this fixed number of moles of BrO_3^- to react. The rate of each reaction is determined by the time required for the color to change, since in Equation 4 the change in concentration is inversely proportional to the time, t. We are mainly concerned with relative rather than absolute rate; therefore, we will take all relative rates as being equal to 1 / t. Fill in the table on the next page, first calculating the relative reaction rate for each mixture.

Name: ______________________________ Section: ____________________

Table 1. Reaction Rate Data

Reaction Mixture	Time (sec) for color change	Relative Rate = 1/t	$[I^-]_{ini}$	$[BrO_3^-]_{ini}$	$[H^+]_{ini}$	Room Temp (°C)
1						
2						
3						
4						
5						

Varying conditions for Mixture 2	Time (sec) for color change	Temp (°C)
Hot: ~ 40 °C		
Cold: ~ 10 °C		
With a catalyst		

The reactant concentrations in the reaction mixture are not those of the stock solutions, since the reagents were diluted by the other solutions. The final volume of the reaction mixture is 25 mL in all cases. We can calculate the concentrations of all reactants in the table above ($M_1V_1 = M_2V_2$).

Determination of the Orders of the Reaction

Given the data in the table, the problem is to find the order for each reactant and the rate constant for the reaction. Since we are dealing with relative rates, we can modify Equation 2 to read as follows:

$$\text{relative rate} = k' [I^-]^m [BrO_3^-]^n [H^+]^p \qquad (5)$$

We need to determine the relative rate constant k' and the orders m, n and p in such a way as to be consistent with the data in Table 1.

The solution to this problem is quite simple once you make a few observations on the reaction mixtures. Each mixture (2 to 4) differs from Reaction Mixture 1 in the concentration of only one species (see table). This means that for any pair of mixtures that includes reaction Mixture 1, there is only one concentration that changes. We can then find the order for the reactant whose concentration was changed.

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Write Equation 5 below for Reaction Mixtures 1 and 2, substituting the relative rates and the initial concentrations of I^-, BrO_3^-, and H^+ from Table 1.

Relative rate 1 = _____________ = k [$]^m$ [$]^n$ [$]^p$

Relative rate 2 = _____________ = k [$]^m$ [$]^n$ [$]^p$

Solve for m, the order of the reaction with respect to the I^-, to two decimal places and then round off to the nearest integer.

m = ________ (2 decimal places) m = ________ (nearest integer)

Apply the same approach to find the value of n, the order of the reaction with respect to the BrO_3^- ion. Show your set-up.

n = ________ (2 decimal places) n = ________ (nearest integer)

Apply the method once again to find p, the order with respect to the H^+ ion. Show your set-up.

p = ________ (2 decimal places) p = ________ (nearest integer)

Having found m, n, and p (nearest integers, the rate law is written as:

rate = ______________________________

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Determination of the Value of Relative Rate Constant

The relative rate constant, k' can be calculated by substitution of m, n, p and the known rates and reactant concentrations into Equation 5. Fill Table 2 below by calculating k for Reaction Mixtures 1 – 4.

Table 2. Reaction Constant, k

Reaction	1	2	3	4	Average	Units of k
k						

Calculations for each k value:

Briefly explain why k should have nearly the same value for each of the Mixtures 1 – 4.

Why? ____

Determination of Relative Rate and Reaction Time for Mixture 5

For Reaction Mixture 5, use k_{ave} from Table 2 above and the appropriate concentrations from Table 1 in Equation 5 to predict (calculate) the relative rate. Then use this value to predict (calculate) the reaction time, t, for Mixture 5. Show your calculations below.

relative $\text{rate}_{predicted}$ __________ $t_{predicted}$ __________ $t_{observed}$ __________

Calculate the percent difference of your reaction times.

% difference ____________

Name: ______________________________ Section: ____________________

The Effect of Temperature on Reaction Rate: The Activation Energy

To find the activation energy for the reaction, transfer the reaction times and temperatures onto Table 3 and fill in the remaining boxes using Mixture 2. Since the reactions at the different temperatures all involve the same reactant concentrations, the rate constants, k, for two different mixtures will have the same ratio as the reaction rates themselves for the two mixtures. This means that in the calculation of E_a, we can use the observed relative rates instead of rate constants.

Table 3. Reaction Rate at Different Temperatures for Mixture 2.

	Room Temp	Hot: ~ 40 °C	Cold: ~ 10 °C
Time (sec) for color change			
Actual Temp (°C)			
Actual Temp (Kelvin)			
1 / Temp (Kelvin^{-1})			
Relative Rate = 1/time			
ln (Relative Rate)			

Use the appropriate values from Table 3 and plot ln(relative rate) vs 1 / T in Microsoft Excel.® Insert a linear trendline and display both the equation and r^2 value of the trendline on the graph. Copy these in the space below. **Make certain to include your Arrhenius plot with this report**.

Trendline Equation: ______________________

r^2 = ________________

The slope of the line equals $-E_a / R$, where R = 8.314 Joules / (mole K). Use this relationship to calculate the activation energy, E_a, for your reaction in kJ/mol.

E_a = ________________ kJ / mol

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The Effect of a Catalyst on Reaction Rate

	Room Temperature Mixture 2	Catalyzed Mixture 2
Time for color to appear (sec)	____________________	____________________

Would you expect the activation energy, E_a, for the catalyzed reaction to be greater than, less than, or equal to the activation energy for the uncatalyzed reaction? Briefly explain.

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Pre-Lab Questions: Iodine Clock Reaction

1. A student studied the clock reaction described in this experiment. She set up a reaction mixture by mixing 10.0 mL of 0.010 M KI, 10.0 mL of 0.0010 M $Na_2S_2O_3$, 20.0 mL of 0.040 M $KBrO_3$ and 10.0 mL of 0.10 M HCl using the procedure given. It took 40.0 seconds for the color to turn blue.

    a. She found the concentration of each reactant in the reacting mixture by realizing that the number of moles of each reactant did not change when that reactant was mixed with the others, but that its concentration did. The volume of the mixture was 50.0 mL. Find the initial concentration of each reactant.

    $[I^-]_{ini}$ = __________ M; $[BrO_3^-]_{ini}$ = __________ M; $[H^+]_{ini}$ = __________ M

    b. What is the relative rate of the reaction (1/time)?

    ____

2. The student repeated the experiment using reaction mixture 1 by mixing 10 mL of 0.010 M KI, 10 mL of 0.0010 M $Na_2S_2O_3$, 10 mL of 0.040 M $KBrO_3$, 10 mL of 0.10 M HCl and 10 mL of H_2O. It took 75 seconds to turn blue.

    Find the initial concentration of each reactant after mixing:_

    $[I^-]_{ini}$ = __________ M; $[BrO_3^-]_{ini}$ = __________ M; $[H^+]_{ini}$ = __________ M

    What is the relative rate of the reaction (1 / time)?

    ____

3. Use the information above to determine the order with respect to BrO_3^-.

    n = ________ (2 decimal places) n = ________ (nearest integer)

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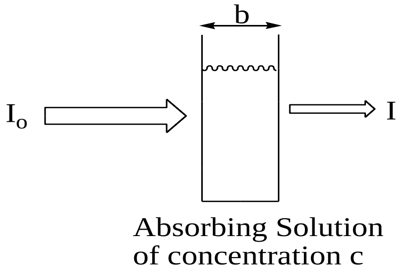
Experiment #6 – Colorimetric Determination of Co^{+2}

Although volumetric and gravimetric mass analyses are commonly used, spectroscopy is the technique most often used for modern chemical analysis. Spectroscopy is the study of electromagnetic radiation emitted or absorbed by a given chemical species. Since the quantity of radiation absorbed or emitted can be related to the quantity of the absorbing or emitting species present, this technique can be used for quantitative analysis. There are many spectroscopic techniques available from X-rays, ultraviolet, infrared, and visible light, to name a few. We will consider one form here which is based on the absorption around visible light.

If a liquid is colored, it is because some component of the liquid absorbs visible light. In a solution, the greater the concentration of the light-absorbing substance, the more light absorbed and the more intense the color of the solution. The quantity of light absorbed by a substance can be measured using a spectrophotometer. The instrument consists of: (1) a source that emits wavelengths of light in a measurable region (i.e. visible light which has wavelengths 400 to 700 nm); (2) a monochromator which selects a given wavelength of light; (3) a sample holder for the solution being measured; and (4) a detector which compares the intensity of incident light I_o to the intensity of light after it has passed through the sample I. When a beam of light passes through a substance, some of the energy is often absorbed by the substance. This causes a decrease in the intensity of the transmitted beam. The ratio I / I_o is called the transmittance, T, a measure of the fraction of light that passes through the sample holder (or cuvette) which contains the absorbing solution. The amount of light absorbed by the solution is given by the absorbance, A, where:

$$A = -\log (I / I_o) = -\log T \qquad (1)$$



$T = I/I_o$

$A = -\log (I/I_o) = -\log T$

$\%T = 100\% T$

The distance, b, the light travels through the solution (in cm) and the concentration, c, of the absorbing species (in mol / L) are represented in the schematic above. A beam of parallel radiation with an intensity is shown before (I_o) and after (I) it has passed through a layer of solution with a measured thickness at a certain concentration. The Beer-Lambert law is the basis for using spectroscopy in quantitative analysis which relates absorbance (A) to the concentration of the absorbing solution (c) and the path length of the cuvette (b). That is:

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$$A = \varepsilon\, b\, c \qquad (2)$$

where ε is the molar absorptivity or the molar extinction coefficient (in L / mol·cm). Each pure substance has its own unique extinction coefficient. Note that during the experiment, the same cuvette should be used for all measurements. With the same cuvette, the path length (b) and the extinction coefficient (ε) remain constant. Therefore, we can mathematically say that ε b = k (constant). If we write the concentration (c) as M for molarity, our new equation becomes:

$$A = k\, M \qquad (3)$$

Once absorption values for different concentrations are obtained, a Beer's law plot of absorbance (vertical axis) versus concentration (horizontal axis) is made. A best-fitting line of the data points is constructed, from which you can determine your equation in slope-intercept form $A = (\varepsilon\, b)\, c + 0$ or $A = k\, M + 0$. By measuring the path length of your cuvette, the extinction coefficient can then be calculated.

In this experiment, there will be three basic tasks to accomplish using the spectrophotometer. First, students will collectively determine the wavelength at which 0.100 M $Co(NO_3)_2$ will absorb best. Next, a standard absorbance curve from which the extinction coefficient can be calculated will be constructed. Finally, an unknown $Co(NO_3)_2$ solution will be analyzed for concentration determination.

Procedure

Part I. Prepare a standard set of $Co(NO_3)_2$ solutions

Using a buret containing the stock solution of 0.100 M $Co(NO_3)_2$ and a 10.00 mL volumetric flask, prepare the following solutions of Co^{2+} by dilution: 0.0800 M, 0.0600 M, 0.0400 M, 0.0200 M.

Part II. Calibrate the Spectrovis

**** If you are the second user of the Spectrovis, you may skip Part II of this procedure and start at Part III Step 1 using the solutions that you prepared in Part I.***

1. Plug the USB cable from the Spectrovis into the PC.
2. Open Logger Pro on the computer.
3. Under Experiment choose Calibrate then Spectrometer 1.
4. Allow the 90 s warmup.
5. Fill the cuvette (up to about ¾) with the blank solution (blank is just deionized water for this experiment). Insert the cuvette into the Spectrovis. Make sure the clear sides are the ones in the light path.
6. Select Finish Calibration.
7. When OK becomes available, select it.
8. The machine is now calibrated and there is no need to re-calibrate unless you exit out of Logger Pro.

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Part III. Determination of Maximum Absorbance Wavelength.

To obtain a ***spectrum and determine λ_{max}***:

1. Discard the blank solution and use the same cuvette for the next steps.
2. Fill your cuvette (up to about ¾) with the stock 0.100 M Co^{2+} solution.
3. Insert the cuvette into the Spectrovis.
4. Select the green button on the computer screen to start collection.
5. Once the spectrum is stabilized (usually takes a second), click stop.
6. To determine the wavelength where absorbance is maximum (λ_{max}), move your cursor over the peak and write down the λ (the reading should be down in the lower left hand corner).

Part IV. Standard Calibration Curve

1. Click the Configure Spectrometer button .
2. Choose the Absorbance vs Concentration option.
3. ON the bottom drop down menu, change from single 10 nm into individual wavelengths.
4. Choose the λ_{max} you determined from the previous step.
5. Click OK.
6. If one or two pop-up window/s appear/s, choose Erase and Continue AND/OR “No” on the next pop-up window.
7. Click collect (green button).
8. Fill (up to about ¾) your cuvette with the stock (0.100 M) Co^{2+} solution and insert the cuvette into the Spectrovis.
9. When the absorbance reading stabilizes (usually takes a second), click KEEP.
10. On the pop-up window, enter the concentration of the sample and click OK. ***DO NOT CLICK STOP.*** You will click STOP in step 13.
11. Discard the solution in the cuvette.
12. Repeat steps 8 through 11 for each of the standard solutions that you prepared. Also, repeat step 8 through 11 for deionized water (a 0.000 M solution of Co^{2+}).
13. When you have measured ***ALL*** of these solutions, Click STOP to end data collection.
14. Click Linear Fit . If a pop-up window appears, choose the “latest run” option.
15. Write down the slope of the line (what is the slope equal to?).

Part V. Determination of Unknown [Co^{2+}]

1. Fill the cuvette with your unknown solution.
2. Insert the cuvette into the Spectrovis.
3. Click collect. If a pop-up window appears, choose “store latest run.”
4. Write down the absorbance (shown on the bottom left corner of the screen).
5. Calculate the concentration of your unknown solution using Beer’s Law.

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Data and Calculations

Wavelength of maximum absorbance is __________________.

$[Co^{+2}]$	Volume of 0.100 M $Co(NO_3)_2$ Stock Solution	ABSORBANCE
0.1000 M	________________________________ mL	____________
0.0800 M	________________________________ mL	____________
0.0600 M	________________________________ mL	____________
0.0400 M	________________________________ mL	____________
0.0200 M	________________________________ mL	____________
0.0000 M	________________________________ mL	____________

UNKNOWN # ____________ ABSORBANCE __________

Path length of the cuvette: _____________

Using Microsoft Excel, plot a graph of absorbance (y) verses concentration (x). Using the graph plotted from your data and the path length of the cuvette, calculate the extinction coefficient.

Extinction Coefficient: ______________

Solve for the concentration on your unknown solution…

(a) $[Co^{+2}]$ ________________(read from graph)

(b) $[Co^{+2}]$ ________________ (calculate from line equation and slope value)

SHOW CALCULATIONS:

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Post-Lab Questions: Colorimetric of Co^{+2}

1. Calculate the transmittance of a solution if its absorbance is 0.352.

2. Calculate the absorbance of a solution if the transmittance is 0.647.

3. The following absorbance values for four solutions with known MnO_4^- concentrations were measured using a spectrophotometer:

Solution	**[MnO_4^-]**	**Absorbance**
1	0.700×10^{-4} M	0.175
2	1.00×10^{-4} M	0.250
3	2.00×10^{-4} M	0.500
4	3.50×10^{-4} M	0.875

A. Using Microsoft Excel, plot a graph of Absorbance vs. Concentration of MnO_4^-. Write the trendline linear equation from the plotted graph.

B. Determine the slope of the graph and include its units.

C. Determine the concentration of an unknown MnO_4^- sample whose absorbance is 0.780.

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D. Using the graph paper, below, construct a graph of Absorbance vs Concentration of MnO_4^-. Draw a linear trendline and determine the equation of the line that you drew. How does this compare to the graph that you made using Excel?

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Experiment #7 – Determination of the Equilibrium Constant for the Formation of $FeSCN^{+2}$

When chemical substances react, the reaction typically does not go to completion. Rather, the system goes to some intermediate state in which both the reactants and products have concentrations which do not change with time at a particular temperature. Such a system is said to be at chemical equilibrium. This condition is expressed in the equilibrium constant, K_c, for the reaction.

In this experiment, we will study the equilibrium properties of the complex ion formation reaction between the iron(III) ion and the thiocyanate ion:

$$Fe^{3+}(aq) + SCN^{-}(aq) \rightleftharpoons FeSCN^{2+}(aq) \qquad (1)$$

When solutions containing Fe^{3+} ion and thiocyanate ion are mixed, Reaction 1 occurs to some extent, forming the $FeSCN^{2+}$ complex ion, which has an orange color. As a result of the reaction, the equilibrium amounts of Fe^{3+} and SCN^{-} will be less than they would have been if no reaction occurred; for every mole of $FeSCN^{2+}$ that is formed, one mole of Fe^{3+} and one mole of SCN^{-} will react. According to our studies, the equilibrium constant expression, K_c, for Reaction 1 is formulated as follows:

$$\frac{[FeSCN^{2+}]}{[Fe^{3+}][SCN^{-}]} = K_c \qquad (2)$$

The value of K_c in Equation 2 is constant at a given temperature. This means that mixtures containing Fe^{3+} and SCN^{-} will react until Equation 2 is satisfied so that the same value of the K_c will be obtained no matter what initial amounts of Fe^{3+} and SCN^{-} were used. Our purpose in this experiment will be to find K_c for this reaction involving several mixtures made up in different ways as well as to show that K_c indeed has the same value in each of the mixtures. The reaction studied is a particularly good one because K_c is of a convenient magnitude, the color of the $FeSCN^{2+}$ ion makes for an easy analysis of the equilibrium mixture, and the reaction reaches equilibrium quickly.

The mixtures will be prepared by combining solutions containing known concentrations of iron(III) nitrate, $Fe(NO_3)_3$, and potassium thiocyanate, KSCN in water. The color of the $FeSCN^{2+}$ ion formed will allow us to determine its equilibrium concentration. Knowing the initial composition of a mixture and the equilibrium concentration of $FeSCN^{2+}$, we can calculate the equilibrium concentrations of the rest of the pertinent species and then determine K_c.

To determine the [$FeSCN^{2+}$] in the equilibrium mixtures, we will use a spectrophotometer, which measures the amount of light absorbed by the orange complex at ***447 nm***, the wavelength at which the complex most strongly absorbs. The absorbance, A, of the complex is proportional to its concentration, M, and can be measured directly on the spectrophotometer. Review the Experiment Six introduction on Beer's Law and procedure on how to calibrate and operate the spectrophotometer. Once the absorbance value is recorded, use the value $\boldsymbol{k = 5.00 \times 10^3}$ to determine the concentration (M) of $FeSCN^{2+}$ for each solution using the equation $\boldsymbol{A = k\,M}$.

Procedure

Label five regular CLEAN, DRY test tubes 1 to 5 and place them in a test tube rack. Use a buret to deliver the appropriate amount of each reactant into a 10.00 mL volumetric flask. Then add enough D.I. water to bring the solution volume to the 10.00 mL mark. Transfer this mixture to the appropriate labeled test tube. Rinse the volumetric flask with D.I. water and prepare the remaining mixtures in similar fashion. *Prepare mixture 5 by adding the* $Fe(NO_3)_3$ *and KSCN directly to the test tube; do not use the volumetric flask.* The reagents to be added for each mixture are summarized:

	1	2	3	4	5
Volume $Fe(NO_3)_3$ solution (mL)	5.00	5.00	5.00	5.00	5.00
Volume KSCN solution (mL)	1.00	2.00	3.00	4.00	5.00
H_2O (mL)	Enough to bring total volume to 10.00 mL				

Mix each solution thoroughly with a glass stirring rod and allow them to reach equilibrium for at least 5 minutes. Be sure to DRY THE STIRRING ROD AFTER MIXING EACH SOLUTION!

Instructions for Using Spectrovis with PC.

a) Plug the USB cable from the Spectrovis into the PC.
b) Open Logger Pro on the computer.
c) Under Experiment choose Calibrate then Spectrometer 1.
d) Allow the 90 s warmup.
e) Fill the cuvette (up to about ¾) with the blank solution (blank is just water for this experiment). Insert the cuvette into the Spectrovis. Make sure the clear sides are the ones in the light path.
f) Select Finish Calibration.
g) When OK becomes available, select it.
h) The machine is now calibrated and there is no need to re-calibrate unless you exit out of Logger Pro.

i) Click the Wave or Configure Spectrometer button .
j) Choose the Absorbance vs Concentration option.
k) ON the bottom drop down menu, change from single 10 nm into individual wavelengths.

- l) Choose the wavelength closest to λ_{max} for the orange complex (447 nm).
- m) Click OK.
- n) If one or two pop-up window/s appear/s, choose Erase and Continue AND/OR "No" on the next pop-up window.
- o) Click collect (green button).
- p) Fill (up to about ¾) your cuvette with one of your five mixtures and insert the cuvette into the Spectrovis.
- q) When the absorbance reading stabilize (usually takes a second), click KEEP.
- r) On the pop-up window, enter the solution number (e.g. 1, 2, 3 etc..).
- s) Discard the solution in the cuvette.
- t) Repeat steps p through s for your other solutions.
- u) When you have measured ***ALL*** of your standard solutions, Click STOP to end data collection. ***DO NOT EXIT OUT OF LOGGER PRO SO THE NEXT USER WILL NOT HAVE TO CALIBRATE THE MACHINE.***
- v) Write down the absorbance values for each of your five solutions on the data table.

**** If you are the second or third user of the Spectrovis, start from step o above (Click the collect (green button)).***

Calculate K_c assuming the reaction: $Fe^{3+}(aq) + SCN^{-}(aq) \rightleftharpoons FeSCN^{2+}(aq)$.

Step 1. Calculate the equilibrium concentration of $FeSCN^{2+}$ from the absorbance using $A = k[FeSCN^{2+}]$, where $k = 5.00 \times 10^3$.

Step 2. Find the new initial concentrations of Fe^{3+} and SCN^{f-} in the mixtures in test tubes 1 through 5 by performing a dilution calculation. Enter the values in the first two columns of the table.

Step 3. Enter the experimentally determined value of $[FeSCN^{2+}]$ at equilibrium for each of the mixtures in column 5 of the table.

Step 4. From the concentration of Fe^{3+} and SCN^{-} initially present in each mixture and the equilibrium concentration of $FeSCN^{2+}$, calculate the concentration of Fe^{3+} and SCN^{-} that remain in each mixture at equilibrium. Enter these results in columns 3 and 4 in the table.

Step 5. Calculate K_c for the reaction for each mixture by substituting values for the equilibrium concentrations of Fe^{3+}, SCN^{-}, and $FeSCN^{2+}$ into Eq. 2.

Name: ______________________________ Section: ____________________

Pre-Lab Questions: Equilibrium Constant Determination for $FeSCN^{+2}$

1. A student mixes 5.00 mL of 2.00×10^{-3} M $Fe(NO_3)_3$ with 5.00 mL of 2.00×10^{-3} M KSCN. She finds that in the equilibrium mixture, the concentration of $FeSCN^{2+}$ is 1.40×10^{-4} M. Find K_c for $Fe^{3+}(aq) + SCN^{-}(aq) \rightleftharpoons FeSCN^{2+}(aq)$.

Step 1. Calculate the initial, diluted concentrations of the Fe^{3+} and SCN^{-} ions in the total of 10.00 ml solution using $M_1V_1 = M_2V_2$

$[Fe^{3+}]$____________ $[SCN^{-}]$____________

Step 2. Use the initial concentrations of the Fe^{3+} and SCN^{-} ions along with the equilibrium concentration of the $FeSCN^{2+}$ ion and the reaction stoichiometry to determine the equilibrium concentrations of Fe^{3+} and SCN^{-}.

	$[Fe^{3+}]$	+	$[SCN^{-}]$	$\leftrightharpoons$	$[FeSCN^{2+}]$
initial	_________		_________		_________
Δ	_________		_________		_________
equil	_________		_________		_________

Step 3. Solve for the value of K_c for the reaction. (Use Eq. 2 and the results of Step 2.)

K_c = _____________

Name: ______________________________ Section: ____________________

Data: Determination of the Equilibrium Constant for the Formation of $FeSCN^{+2}$

Mixture	Vol. of 2.00 × 10^{-3} M $Fe(NO_3)_3$ (in mL)	Vol. of 2.00 × 10^{-3} M KSCN (in mL)	Vol. of H_2O (in mL)	Absorbance
1	5.00	1.00	4.00	__________
2	5.00	2.00	3.00	__________
3	5.00	3.00	2.00	__________
4	5.00	4.00	1.00	__________
5	5.00	5.00	0.00	__________

Mixture	Initial Concentrations AFTER MIXING		Equilibrium Concentrations			
	$[Fe^{+3}]$	$[SCN^-]$	$[Fe^{+3}]$ +	$[SCN^-]$	⇋ $[FeSCN^{+2}]$	K_c
1	__________	__________	__________	__________	__________	__________
2	__________	__________	__________	__________	__________	__________
3	__________	__________	__________	__________	__________	__________
4	__________	__________	__________	__________	__________	__________
5	__________	__________	__________	__________	__________	__________

Hint: Set up each mixture as shown in the pre-lab *Reaction-Initial-Change-Equilibrium*

Average K_c ____________

Name: ________________________________ Section: ____________________

Post-Lab Questions: Determination of the Equilibrium Constant for the Formation of $FeSCN^{+2}$

1. Are the K_c values on the previous page consistent? If not, suggest a reason for any large differences.

2. In carrying out this analysis, we made the assumption that the reactants were reacting as a 1:1 mole ratio, as given by Equation 1. There is no inherent reason why the reaction might not have been a 1:2 mole ratio:

$$Fe^{3+}(aq) + 2\ SCN^{-}(aq) \rightleftharpoons Fe(SCN)_2^{+}(aq) \qquad (3)$$

   a. Fill in the equilibrium values in the chart below using your experimental data and this new reaction ratio:

Reaction	**$Fe^{+2}(aq)$ +**	**$2\ SCN^{-}(aq)$ $\rightleftharpoons$**	**$Fe(SCN)_2^{+}(aq)$**
test tube 1 mixture at equilibrium			
test tube 5 mixture at equilibrium			

   b. Calculate the value of K_c using the data from the test tube 1 mixture, assuming that the reaction is actually the one shown in equation 3.

   c. Calculate the value of K_c using the data from the test tube 5 mixture, assuming that the reaction is actually the one shown in equation 3.

   d. Compare the K_c values that you calculated in parts a and b above. Are they consistent? Do you think Reaction 3 is occurring?

Name: ________________________________ Section: ____________________

Experiment #8 – Solubility, K_{sp}, Common Ion Effect and $\Delta H_{soln}°$ of Potassium Hydrogen Phthalate

In this experiment you will determine the solubility and solubility product constant, K_{sp}, of Potassium Hydrogen Phthalate ($KHC_8H_4O_4$ abbreviated as KHP) at various temperatures. Equation 1 below shows the solubility product equation of KHP.

$$KHC_8H_4O_{4(s)} \overset{H_2O}{\Longleftrightarrow} K^+_{(aq)} + HC_8H_4O_4{}^-_{(aq)} \qquad (1)$$

The K_{sp} expression associated with this reaction is given in equation 2 below.

$$K_{sp} = [K^+]\,[HC_8H_4O_4{}^-] \qquad (2)$$

Because of the 1:1 mole ratio of $K^+ : HC_8H_4O_4{}^-$, we need only determine the concentration of one of these to calculate K_{sp} assuming that there is no source of these ions other than KHP. ***Also, note that the solubility of KHP is equal to*** $[HC_8H_4O_4{}^-]$***.*** The acid dissociation of $HC_8H_4O_4{}^-$ can be treated as negligible as its K_a is around 4×10^{-6}. This K_a is four to five magnitudes smaller than the K_{sp} of KHP. KHP is a well known primary standard used in standardizing base solutions such as NaOH (aq). $HC_8H_4O_4{}^-$ reacts with OH^- (from NaOH) as described in equation 3 below.

$$HC_8H_4O_4{}^-_{(aq)} + OH^-_{(aq)} \rightarrow C_8H_4O_4{}^{2-}_{(aq)} + H_2O_{(l)} \qquad (3)$$

In this experiment you will use saturated solutions of KHP at different temperatures. You will titrate an aliquot of the supernate (liquid above the solid) with a standard NaOH solution to determine $[HC_8H_4O_4{}^-]$ of each saturated solutions. You will then calculate K_{sp} at each temperature. Once K_{sp} at each temperature is obtained, you will use equation 4a to obtain the standard enthalpy of dissolution $\Delta H°_{soln}$ and the standard entropy of dissolution, $\Delta S°_{soln}$.

$$\ln K_{sp} = \frac{-\Delta H^o_{soln}}{R}\left(\frac{1}{T}\right) + \frac{\Delta S^o_{soln}}{R} \qquad (4a)$$

Note that equation 4a is in the form of an equation of a straight line (y = mx + b). Equation 4a is similar to the Arrhenius equation in the kinetics experiments that used specific rate constants (k) and temperature (T) to solve for activation energy (E_a). A plot of ln K_{sp} vs 1/T (in Kelvin) will allow you to solve for $\Delta H°_{soln}$ and $\Delta S°_{soln}$. R is the gas constant 8.314 J mol^{-1} K^{-1}. $\Delta H°_{soln}$ is the heat associated with reaction 1 at constant pressure. $\Delta S°_{soln}$ is the change in entropy of reaction 1. Entropy is a measure of energy dispersal (ways of being or randomness). A state in which a given amount of energy is more highly dispersed has more entropy than a state in which the same energy is more highly concentrated. If you only had two sets of K_{sp} and T, you could use equation 4b to calculate $\Delta H°_{soln}$.

$$\ln \frac{K_2}{K_1} = \frac{\Delta H^o_{soln}}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \qquad (4a)$$

Additionally, you will observe the common ion effect by comparing $[K^+]$ or $[HC_8H_4O_4^-]$ between a saturated solution of KHP and a solution that is also saturated with KHP but was prepared using 0.50 M KCl (both at room temperature). Note that common ion effect states that the solubility of a solid decreases when a common ion is present in the solution. Therefore, $[K^+]$ or $[HC_8H_4O_4^-]$ should be lower in a saturated solution where K^+ (a common ion from KCl) is present. You will be asked to calculate % decrease in solubility as given in equation 5 below.

$$\%\ \text{decrease} = \left(\frac{[HC_8H_4O_4]_{\text{water}} - [HC_8H_4O_4]_{\text{sat'd KCl}}}{[HC_8H_4O_4]_{\text{water}}}\right) \times 100\% \qquad (5)$$

You will calculate the room temperature K_{sp} in both water and with 0.50M KCl-saturated solutions. You will determine the consistency of your two room temperature K_{sp} values by calculating the % difference between them using equation 6. Ideally you will see that even when there is a common ion present, the value for K_{sp} should remain the same or constant (0% difference) between the two room temperature saturated solutions (water and with 0.50M KCl-saturated solution).

$$\%\ \text{difference} = \left(\frac{Ksp_{\text{water}} - Ksp_{\text{sat'd KCl}}}{Ksp_{\text{average}}}\right) \times 100\% \qquad (6)$$

Procedure

1. Transfer the indicated amount of the saturated KHP solution into an Erlenmeyer flask. Make sure that you withdraw from the top portion of the flask and take care not to agitate the solid that has settled on the bottom of the flask. If you see any KHP crystals floating on the surface, dip a small piece of clean dry paper towel into the liquid to remove the crystals.

2. Add about 20 mL deionized water and 2 drops of phenolphthalein indicator to the flask. Titrate the saturated KHP solution with standardized NaOH of known molarity until a persistent pink color is established.

3. Repeat steps above for the saturated solutions (in water) at two other temperatures and the one at room temperature with 0.50 M KCl.

Name: ________________________________ Section: ____________________

Pre-Lab Questions

1. A student withdraws 3.00 mL of supernate from a saturated solution of $KHC_8H_4O_4$ at room temperature (~ 22 ^oC). This sample was titrated to the phenolphthalein endpoint and 12.85 mL of 0.0997 M NaOH was required.

   a. Calculate moles of $HC_8H_4O_4^-$ in the 3.00 mL aliquot.

   b. Calculate the $[HC_8H_4O_4^-]_{equil}$ (a.k.a. solubility).

   c. What is the value for $[K^+]_{equil}$?

   d. Calculate K_{sp} at 22 ^oC.

2. A similar titration as in #1 above was done for a saturated solution of KHP in 0.500 M KCl also at room temperature. The withdrawn supernate was 5.00 mL (instead of 3.00 mL). The volume of titrant (0.0997 M NaOH) used was 8.35 mL.

   a. Calculate the new solubility in 0.500 M KCl.

b. Calculate the % decrease in solubility by using equation 5.

3. Two more titrations like the one in #1 above were done at two other temperatures and a plot of ln K_{sp} vs 1/T (in Kelvin) was constructed and yielded the trendline equation below. Calculate $\Delta H°_{soln}$ (**kJ/mol**) and $\Delta S°_{soln}$ (**kJ/K·mol**).

   $y = -4480\,x + 13.8$

$\Delta H°_{soln}$ = _________________ **kJ/mol** $\Delta S°_{soln}$ = __________________ **kJ/K·mol**

Name: ___________________________________ Section: ______________________

Data and Calculations

Concentration of standard NaOH: ______________ M

Sample of sat'd KHP	Temperature (Kelvin)	Vol KHP acid (mL)	Vol NaOH base (mL)	$[HC_8H_4O_4^-]_{eq}$ ***(solubility)*** (M)	$[K^+]_{eq}$ (M)	K_{sp}
At room temp						
warmer						
colder						
At room temp with 0.50M KCl						

Show sample calculations for $[HC_8H_4O_4^-]_{eq}$, $[K^+]_{eq}$ and K_{sp} below:

Prepare an Excel® chart filling in K_{sp} and T values. Have the computer program calculate $\ln(K_{sp})$ and 1 / T for the 4 sets of data.

Plot a scatter graph with $y = \ln(K_{sp})$ values and $x = 1/T$ (Kelvin) and display the linear trendline equation of your plot. Attach your graph.

Write out the linear trendline equation _________________________________

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Post-Lab Questions:

1. Based on your results at room temperature for saturated KHP in water, calculate the solubility of KHP in g / L.

2. What will happen to the solubility of saturated KHP at room temperature if NaOH is added to the solution? (increase, decrease or no change)? Explain your reasoning.

   Additionally, how would adding NaOH affect the K_{sp} of KHP at room temperature?

3. Calculate the % decrease in solubility at room temperature between saturated KHP and saturated KHP in 0.50M KCl according to equation 5.

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4. Calculate the % difference in K_{sp} at room temperature, equation 6. Based on a 10% acceptable difference, are your K_{sp} values in water and in KCl the same? If they are not within 10% of each other, propose a specific error that might have caused this significant difference in K_{sp}.

5. Plot ln K_{sp} vs 1 / T (Kelvin) and determine the linear trendline equation of your plot. Attach your graph.

   Linear Trendline Equation: ____

6. Calculate the ΔH°_{soln} and ΔS_{soln}^o for the dissolution of solid KHP from the trendline equation of your scatter graph; $\ln(K_{sp})$ vs 1 / T(Kelvin). R is the gas constant 8.314 J mol^{-1} K^{-1}

   ΔH°_{soln} = _________________ **kJ / mol** ΔS°_{soln} = __________________ **kJ / K·mol**

Name: ________________________________ Section: ____________________

Experiment #9 – pH Measurements, Buffers and Determination of the Equivalent Mass and K_a of an Unknown Weak Acid

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One of the more important properties of an aqueous solution is its concentration of hydrogen ion. The H^+ or H_3O^+ ion has great effect on the solubility of many inorganic and organic species, on the nature of complex metallic cations found in solutions, and on the rates of many chemical reactions. It is important that we know how to measure the concentration of hydrogen ion and understand its effect on solution properties.

For convenience, the concentration of H^+ ion is frequently expressed as the pH of the solution rather than as molarity. The pH of a solution is related to $[H^+]$ by the following equations:

$$\mathrm{pH} = -\log [H^+] \qquad \text{and} \qquad [H^+] = 10^{-\mathrm{pH}} \qquad (1)$$

Examples: When $[H^+]$ is 1×10^{-4} moles per liter, the pH of the solution is 4. When the $[H^+]$ is 5×10^{-2} M, the pH is 1.3.

Basic solutions can also be described in terms of pH. In aqueous solutions, the following equilibrium relationship exists:

$$[H^+]\,[OH^-] = K_w = 1 \times 10^{-14} \text{ at } 25\ ^\circ\mathrm{C} \qquad (2)$$

In pure water, $[H^+] = [OH^-]$, so by equation 2, $[H^+] = 1 \times 10^{-7}$ M. Therefore, the pH of pure water is ideally 7. Solutions in which $[H^+] > [OH^-]$ are said to be acidic and will have a $\mathrm{pH} < 7$; if $[H^+] < [OH^-]$, the solution is basic and its $\mathrm{pH} > 7$.

In part 1A of this experiment, you will determine the approximate pH of a liquid unknown solution by using several colorful acid-base indicators and comparing to various buffer solutions. In part 1B you will determine the pH, K_a and % dissociation of several concentrations of acetic acid. Part 2 of the experiment involves predicting the pH of several salt solutions, verifying your predictions using a pH meter, and then writing out hydrolysis equations. In part 3, you will prepare a buffer by half-neutralizing a solution of an unknown weak acid with the strong base NaOH and measure its pH. From the known pH at half equivalence, you will calculate the K_a of the weak acid and then test the buffer's ability to resist change in pH as compared with tap water. In part 4 you will determine the gram equivalent mass (GEM) of a solid unknown acid by titration with a standardized strong base (NaOH). In Part 5 you will use the computerized LabQuest Mini drop counter and pH meter to construct a weak acid/strong base titration curve. From this titration curve data, you will be able to calculate both the gram equivalent mass and the value of the equilibrium constant for the dissociation of the acid, K_a.

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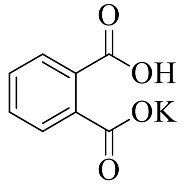
Acids are substances that contain ionizable hydrogen atoms within the molecule. Strong acids ionize completely, weak acids partially. The value of K_a, the equilibrium constant for the dissociation of the acid, is an indication of the strength of the acid. An acid may contain one or more ionizable hydrogen atoms in the molecule. The gram equivalent mass of an acid is the molecular mass divided by the number of ionizable hydrogen atoms in a molecule. For example, hydrochloric acid, HCl, contains one ionizable hydrogen atom; the molecular mass is 36.45 g/mole HCl, and the equivalent mass is also 36.45 g/mole H^+. Sulfuric acid, H_2SO_4, contains two ionizable hydrogen atoms; the molecular mass is 98.07 g/mole H_2SO_4, yet the equivalent mass is 49.04 g/mole H^+. Thus, 36.45 g of HCl or 49.04 g of H_2SO_4 would provide you with one mole of H^+ ions.

The equivalent mass may be determined by titrating an acid with a standardized solution of NaOH. Since one mole of NaOH reacts with one mole of hydrogen ion, at the equivalence point, the following relation holds:

$$V_b \text{ x } M_b = \text{moles base} = \text{moles } H^+$$
$$GEM_a = \text{grams acid/moles } H^+$$

where V_b is the volume of base in liters, M_b is the molarity of base, grams acid is the mass of acid used, and GEM_a is the gram equivalent mass of the acid.

The concentration of the NaOH solution must be accurately known. To “standardize” the NaOH (that is, to find its exact molarity so it becomes a secondary standard), the NaOH is titrated against a solid acid, potassium hydrogen phthalate, sometimes abbreviated KHP (shown below). This acid is chosen because it possesses qualities of a primary standard which include a relatively large molar mass, high purity, unreactive with the atmosphere, one invariable reaction, and soluble in the chosen solvent. Other advantages of using KHP include its affordability, and it is relatively nontoxic compared to other possible choices. Sodium hydroxide cannot be used as a primary solid because it reacts with the atmosphere so it does not remain pure, and it has a relatively low molecular weight. The titration is thus followed using phenolphthalein as an indicator.

KHP or  or $KHC_8H_4O_4$

A weak acid/strong base titration curve of pH versus mL of NaOH has four distinct areas; a) weak acid at zero ml, b) buffer zone, c) salt at equivalence pt, and d) strong base beyond equivalence. There should be a significant change in pH in the vicinity of the equivalence point. Note that the equivalence point will probably NOT be at pH 7, but will be on the basic side.

The equilibrium equation and the corresponding expression are given below.

$$HA(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + A^-(aq) \quad (3)$$
$$K_a = [H_3O^+][A^-] / [HA] \quad (4)$$

When the acid is HALF neutralized, [HA] = [A^-], so these terms cancel in the equation (4), and K_a = [H_3O^+]. Therefore, when the acid is half-neutralized, pH = pK_a. The value of the equilibrium constant for the dissociation of the acid can be determined from the half equivalence point.

The point where pH is equal to pK_a can be found from the graph. Refer to Figure 1.

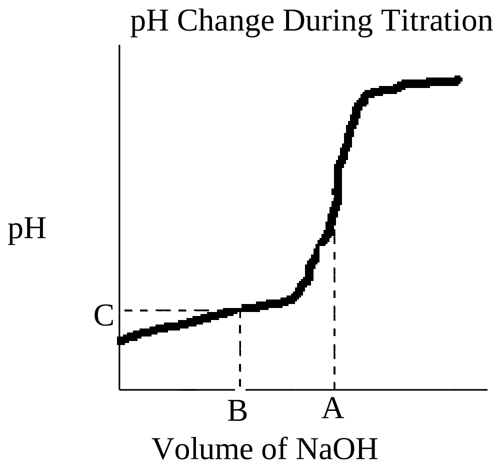


Figure 1. Titration of a Monoprotic Weak Acid with Sodium Hydroxide

In this graph, A = Volume NaOH at equivalence point; B = ½ volume of A or the volume when half-neutralized; and C = pH when half-neutralized, or pK_a.

Safety / Caution: Acids and bases can irritate your skin and eyes. Wear safety goggles during the entire lab. Acid powder spilled on clothing may react with laundry soap (bases) and can cause some holes or torn fibers on clothing.

Procedure

Part 1A: pH of Unknown and Buffer Solutions

1. Obtain a liquid unknown solution and place a drop of the solution onto a small strip of pH paper.

2. Estimate the pH of the liquid unknown solution by comparing the colors of the pH test strip with the color chart on the plastic box of the test strips. The precision of the scale on the box of the pH paper is about ± 1. So note your estimate in a 3-pH unit range (e.g. estimate pH = 7 – 9).

3. Obtain a spot plate. Place 5 drops of your unknown into 3 wells as seen in Figure 2 below. Obtain three buffer solutions that correspond to your 3-pH unit estimate of your unknown. Place 5 drops of each buffer solutions into 3 wells as indicated in Figure 2.

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4. Choose about three indicators that have a useful range that overlaps with most or some of your 3-pH unit range estimate of your unknown. For each indicator, place a drop of one indicator in a well with the unknown and one in each of the three different buffers as seen in Figure 2. Record the colors of each well on the table in Part 1 of Data and Calculations.

5. Use the pH values of the buffers to improve your estimate of the pH of your unknown by looking for the best color-match between your unknown and the buffers. Estimate the pH to the nearest 0.5 pH unit. Record this pH in the space provided below the table in Part 1 of Data and Calculations.

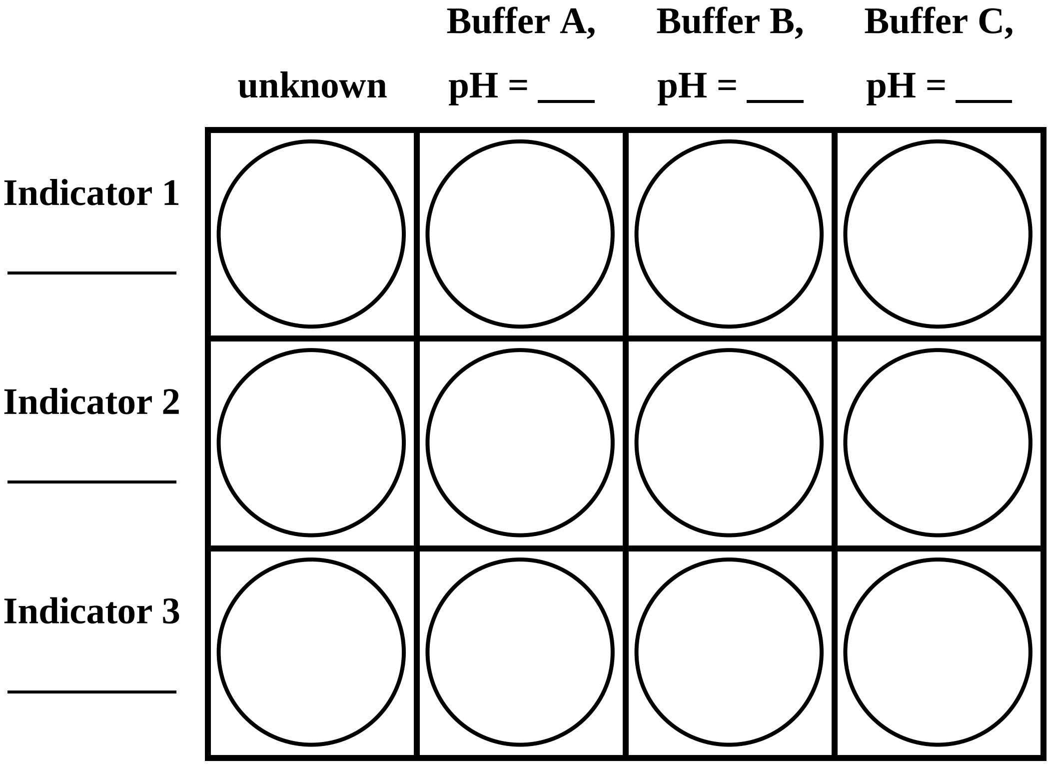

Figure 2. Schematic of first spot plate solution distribution

<u>Part 1B</u>: pH of Acetic Acid Solutions.

1. Record the pH of 1.0 M, 0.10 M and 0.010 M HAc (acetic acid) solutions by reading the three pH meters set up for the class.

2. Calculate the K_a and % dissociation of HAc using these three pH measurements. Show your calculations in part 1 of the Data and Questions section below.

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Part 2: pH of Salt Solutions

1. For the six salt solutions in the Data Sheet, estimate the pH as acidic, neutral, or basic. Record your predictions before proceeding to #2!

2. Once your predictions are complete, read the pH meters immersed in the salt solutions and record the actual pH values on your data sheet. How closely do your predictions correlate with the actual experimental results? Make corrections if needed. Write the molecular equation, complete ionic equation, and net ionic equation for each of the salt solutions in the Data Sheet.

Part 3: Determination of K_a of an Unknown Weak Acid and Properties of a Buffer

1. Obtain a solid sample of an unknown acid. Dissolve approximately 1 g of the unknown sample in 50 mL D.I. water in a 250-mL Erlenmeyer flask. Pour all of the solution into a graduated cylinder read the volume and pour back half of the volume into the original Erlenmeyer flask. Save the other half in the graduated cylinder for step 3 below.

2. Add 2 drops of phenolphthalein indicator into the Erlenmeyer flask and titrate it with the standardized NaOH solution. Volume readings need not be taken here. As the endpoint approaches, add the titrant drop by drop until the solution has a permanent pale pink color.

3. Add the other half of the solution that you set aside (in a graduated cylinder) into the titrated solution in the Erlenmeyer flask. Swirl the solution well. ***This solution is now HALF-NEUTRALIZED.***

4. Rinse the pH meter well over a beaker with D.I. water each time you will measure a new pH value. NEVER wipe the electrode with a paper towel; this can damage the probe. Now measure the pH of the half-neutralized solution using the pH meter. From the observed pH, calculate the K_a of your unknown weak acid using the space provided in part 3 of the Data and Questions section below.

5. The half-neutralized solution is a buffer. Place 20 mL of this buffer in a 100 mL beaker. Measure the initial pH of this 20 mL buffer transferred to a clean 100 ml beaker, add 5 drops of 0.1 M HCl and mix thoroughly. Measure the pH of the resulting solution. Place 20 mL of tap water in another clean 100 mL beaker. Measure the initial pH of this 20 mL tap water in another clean 100 mL beaker. Add 5 drops of 0.1 M HCl into the tap water, mix thoroughly and measure the pH of this acidic solution.

6. Repeat step 5 above, this time adding 0.1 M NaOH (instead of 0.1 M HCl) into a clean beaker containing a new sample of 20 mL of the buffer (half neutralized solution) and a new sample of 20 mL tap water.

Name: ______________________________ Section: ____________________

Part 4: Determination of the Equivalent Mass of an Unknown Acid

1. Accurately weigh a sample of your solid unknown acid (the appropriate mass is written on the unknown container). *Make sure to record the unknown number and mass*.
2. Dissolve the sample in 30 mL D.I. water and titrate it to the phenolphthalein end point.
3. Repeat one more time. Choose a mass for the second sample so that the volume of NaOH needed will be about 15 mL.
4. Calculate the gram equivalent mass of your sample.

Part 5: Determination of the GEM and K_a of an Unknown Acid from a Titration Curve

1. In a 150 ml beaker, weigh and record a sample of your acid that requires about 15 mL of base to reach the equivalence point. *[(acid mass/base volume)$_{part\ 4}$ x15]*
2. Dissolve the sample in approximately 50 mL D.I. water.
3. Set up the computerized LabQuest Mini drop counter and pH meter to construct a weak acid/strong base titration curve. Rinse the pH meter well with D.I. water. NEVER wipe the electrode with a paper towel; this can damage the probe. The magnetic stirrer is attached to the pH probe.
4. Check and set the drop calibration to the correct number of drops per mL: Click on experiment/calibrate/drops per ml and change if necessary to match the drops stated on the dropper instrument containing the standardized NaOH. Verify that the volume of NaOH in the dropper is sufficient.
5. Place your 150 mL beaker on a magnetic stirrer. Insert the pH electrode so it is submerged in your acid solution, you may need to add more water. Turn on the magnetic stirrer. Caution: DO NOT TURN ON HEAT. Line up the dropper so that each drop will pass through the infrared sensor and be measured. Set the experiment to collect. Slowly start drops falling and check to see if they are measured and recorded. Continue the titration curve until about 5 mL beyond the equivalence point. The LabQuest Mini will record the volume of base for each drop and pH of the solution during the titration.
6. Stop the collection and click on analyze/examine. Move the mouse curser to the equivalence point on the graph and record the volume and pH at equivalence. Similarly, record the half equivalence volume and the half-equivalence pH.
7. Save the graph by taking a picture or sending it to your email as an Excel® file or pdf.
8. From the graph, determine the pK_a of the acid, that is, the pH where the acid is half-neutralized. Calculate the K_a value of your acid.
9. Determine the volume of NaOH needed to reach the equivalence point from the graph and with your recorded mass of the acid used, determine the gram equivalent mass of your acid. Calculate the average of all three of the gram equivalent mass values from parts 4 and 5.

Name: ___________________________________ Section: ______________________

Pre-Lab Questions: Buffers and Determination of Equivalent Mass and K_a of an Unknown Acid

1. What is the equivalent mass of each of the following acids?
   1. $HC_2H_3O_2$
   2. $KHCO_3$
   3. H_2SO_3
   4. H_3PO_4
2. It is found that 24.6 mL of 0.116 M NaOH is needed to titrate 0.293 g of an unknown acid to the phenolphthalein end point. Calculate the equivalent mass of the acid.

Name: ________________________________ Section: ____________________

Data and Questions

Part 1A: pH of Unknown and Buffer Solutions

Enter in the appropriate space the name of the indicator used, the observed color of unknown after addition of the indicator, and the estimated pH value from the pH paper for the unknown.

Liquid Unknown #: _________

pH paper estimate: ________________ (3-pH unit range)

Indicator Used	Color of Unknown	Color of Buffer pH = _______	Color of Buffer pH = _______	Color of Buffer pH = _______

Estimate pH based on matching of colors = ___________ (within 0.5 pH unit)

Part 1B: pH of Acetic Acid Solutions

(<u>Note</u>: HAc = $HC_2H_3O_2$*, acetic acid).*

	1.0 M $HC_2H_3O_2$	0.10 M $HC_2H_3O_2$	0.010 M $HC_2H_3O_2$
pH			
K_a			
% dissociation			

SHOW YOUR CALCULATIONS ON THE NEXT PAGE.

Name: ________________________________ Section: ____________________

1.0 M $HC_2H_3O_2$ (aq):

0.10 M $HC_2H_3O_2$ (aq):

0.010 M $HC_2H_3O_2$ (aq):

Name: ___________________________________ Section: ______________________

Part 2: pH of Salt Solutions

1. PREDICT whether each of the salt solutions below is expected to be acidic, neutral, or basic:

$NaCl$ __________ $NaC_2H_3O_2$ __________ Na_2CO_3 ____________

NH_4Cl _________ KNO_3 _____________ $ZnCl_2$ ______________

2. Using the pH meter immersed in each salt solution, determine the actual pH:

$NaCl$ __________ $NaC_2H_3O_2$ __________ Na_2CO_3 ____________

NH_4Cl _________ KNO_3 _____________ $ZnCl_2$ ______________

3. Write balanced MOLECULAR, IONIC, and NET-IONIC equations for the hydrolysis reactions of each salt solution. From the net-ionic equation, verify that the reaction is acidic, neutral or basic.

A. $NaCl(aq)$:

Molecular:

acidic
neutral
or
basic?

Ionic:

_____ Net-Ionic:

B. $NaC_2H_3O_2(aq)$

Molecular:

Ionic:

_____ Net-Ionic:

Name: ___________________________________ Section: ______________________

C. $Na_2CO_3(aq)$

Molecular:

Ionic:

_____ Net-Ionic:

D. $NH_4Cl(aq)$

Molecular:

Ionic:

_____ Net-Ionic:

E. $KNO_3(aq)$

Molecular:

Ionic:

_____ Net-Ionic:

F. $ZnCl_2(aq)$

Molecular:

Ionic:

_____ Net-Ionic:

Name: ________________________________ Section: ____________________

Part 3: Determination of K_a and Properties of a Buffer

Solid Unknown Number: ____________

1. Original pH of the half neutralized solution: ___________

2. Calculate K_a of the Weak acid:

3. Fill in table:

	tap water (original pH)	tap water (pH after)	Buffer (original pH)	Buffer (pH after)
addition of 0.1 M HCl				
addition of 0.1 M NaOH				

4. How does the table above show that the half-neutralized solution is indeed a buffer?

5. Using the data on your table above, comment on the buffering ability of your half-neutralized solution in comparison to the tap water.

6. Comment on the comparison between adding a strong acid vs a strong base to your buffer solution (i.e. is this solution more resistant to an increase or a decrease in pH?).

Name: ________________________________ Section: ____________________

Part 4: Determination of the Equivalent Mass of an Unknown Acid

Given: ___________ M NaOH

Fill in the table below

Sample	Mass unknown acid (g)	Volume NaOH used (mL)	Volume NaOH used (L)	Mol NaOH equal to Mol H^+	Gram Equivalent Mass of Acid (g/mol H^+)
Trial 1					
Trial 2					
					Average GEM: _________ g/mol H^+

Show sample calculations below

Part 5: Determination of the K_a and Equivalent Mass of an Unknown Acid using LabQuest Mini

Use the same unknown sample as part 4. Solid Unknown Number: _______

1. Determine the approximate mass desired to reach the equivalence point in approximately 15 ml of NaOH added.

   Approximate mass to use = (mass of acid/volume of base)$_{part\ 4}$ x 15ml desired

2. Mass accurately weighed into a clean, dry 150 ml beaker. ____________

3. Using the graph, determine the volume and pH of titrant at equivalence point.

   Volume ______________ pH ___________

4. Using the graph, determine the volume and pH at the half-equivalence point.

   Volume ______________ pH ___________

Name: ________________________________ Section: ____________________

5. Solve for the pK_a, K_a, and gram equivalent mass of your unknown acid using the data collected in part 5.

   Unknown #: _______

6. Calculate the average of all three GEM that you determined (two from part 4 and one from part 5).

7. Why is the equivalence point NOT at pH 7?

8. Identify the following areas on the weak acid/strong base titration curve.
   A) Weak acid
   B) Buffer zone
   C) Equivalence point, salt
   D) Strong base zone
   E) Half equivalence point

Name: ______________________________ Section: ____________________

Post-Lab Questions: Buffers and Determination of Equivalent Mass and K_a of an Unknown Acid

1. A buffer was prepared by mixing 50.0 mL of 0.10 M HX and 25.0 mL of 0.10 M NaOH. The K_a of HX is 1.5×10^{-6}. Calculate the pH of this buffer.

2. The following values were experimentally determined for the titration of 0.145 g of a weak acid with 0.100 M NaOH:

Volume of NaOH, mL	**pH**
0.0	2.88
5.0	4.15
10.0	4.58
12.5	4.76
15.0	4.93
20.0	5.36
24.0	6.14
24.9	7.15
25.0	8.73
26.0	11.29
30.0	11.96

A. Construct a titration curve (pH vs Volume of NaOH).

B. Examine the graph for the required volume to reach the equivalence point?

C. Examine the graph and state the pH at the half-equivalence point?

D. Determine the K_a of the acid.

E. Calculate the gram equivalent mass of the acid.

Name: ___________________________________ Section: ______________________

3. The following acid-base indicators are available to indicate the end point of this weak acid/strong base titration. Which of them would be most appropriate? Explain.

Indicator	**Color Change**		**pH Transition**
	Acid Form	**Base Form**	
Bromphenol blue	yellow	blue	3.0-5.0
Bromthymol	blue	blue	6.0-7.6
Thymol blue	yellow	blue	8.0-9.6

Name: ________________________________ Section: ____________________

Experiment #10 – Qualitative Analysis of Group I Cations

Qualitative analysis is a branch of analytical chemistry that identifies particular substances in a given sample of material. In the analysis of inorganic substances, this branch involves the analysis of both metallic constituents as cations and nonmetallic constituents as anions. Qualitative analysis has remained an important part of the laboratory experience in general chemistry for a number of years even though such analytical methods have been replaced by sophisticated instrumental methods for practical analysis.

In this experiment, you will analyze a known solution that contains all the Group I cations—silver, lead (II), and mercury(I)—and an unknown solution to determine which ions are present and which are absent.

The chlorides of Ag^+, Pb^{2+}, and Hg_2^{2+} are all insoluble in cold water. They can be removed as a group from solution by the addition of HCl. The reactions that occur are simple precipitations and can be represented by the equations:

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s) \tag{1}$$

$$Pb^{2+}(aq) + 2\,Cl^-(aq) \rightarrow PbCl_2(s) \tag{2}$$

$$Hg_2^{2+}(aq) + 2\,Cl^-(aq) \rightarrow Hg_2Cl_2(s) \tag{3}$$

It is important to add enough HCl to ensure complete precipitation, but not too large an excess. In concentrated HCl solution, these chlorides tend to dissolve, producing chloro-complexes such as $AgCl_2^-$.

Lead (II) chloride is separated from the other two chlorides by heating with water. The K_{sp} for $PbCl_2$ greatly increases with temperature, favoring less solid and more ions, therefore the $PbCl_2$ dissolves in hot water:

$$PbCl_2(s) \rightarrow Pb^{2+}(aq) + 2\,Cl^-(aq) \tag{4}$$

Once Pb^{2+} has been put into solution, we can check for its presence by adding a solution of K_2CrO_4. The chromate ion, CrO_4^{2-}, gives a yellow precipitate with Pb^{2+}:

$$Pb^{2+}(aq) + CrO_4^{2-}(aq) \rightarrow \underset{\text{yellow}}{PbCrO_4(s)} \tag{5}$$

The other two insoluble chlorides, AgCl and Hg_2Cl_2, can be separated by adding aqueous ammonia. Silver chloride dissolves, forming the complex ion $Ag(NH_3)_2^+$:

$$AgCl(s) + 2\ NH_3(aq) \rightarrow Ag(NH_3)_2^+(aq) + Cl^-(aq) \qquad (6)$$

Ammonia also reacts with Hg_2Cl_2 via a rather unusual oxidation-reduction reaction. The products include finely divided metallic mercury, which is black, and a compound of formula $HgNH_2Cl$, which is white:

$$\underset{\text{white}}{Hg_2Cl_2(s)} + 2\ NH_3(aq) \rightarrow \underset{\text{black}}{Hg(l)} + \underset{\text{white}}{HgNH_2Cl(s)} + NH_4^+(aq) + Cl^-(aq) \qquad (7)$$

As this reaction occurs, the solid appears to change color, from white to black or grey.

The solution containing $Ag(NH_3)_2^+$ needs to be further tested to establish the presence of silver. The addition of a strong acid (HNO_3) to the solution destroys the complex ion and reprecipitates silver chloride. We may consider that this reaction occurs in two steps:

$$\begin{array}{lr}
Ag(NH_3)_2^+(aq) + 2\ H^+(aq) \rightarrow Ag^+(aq) + 2\ NH_4^+(aq) & \\
Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s) & \\
+ & \\
\hline
Ag(NH_3)_2^+(aq) + 2\ H^+(aq) + Cl^-(aq) \rightarrow \underset{\text{White}}{AgCl(s)} + 2\ NH_4^+(aq) & (8)
\end{array}$$

Name: ______________________________ Section: ____________________

Procedure

Wear your safety glasses while performing this experiment. Lead and mercury salts are toxic, and chromates are known to be carcinogenic. Silver ion is corrosive and leaves a black stain on the skin. Make certain to wash your hands thoroughly when you leave the laboratory. Work with two separate solutions in test tubes, (1) a known solution that contains all three ions and (2) a separate test solution with unknown ions. Label your solutions and your stirring rods well with different colored tape to avoid cross contamination between your known and unknown samples.

1. Precipitation of Group I Ions. Add 2 drops of 6 M HCl to 1 mL of each test solution in separate small test tubes. Centrifuge the solution, being careful to balance the centrifuge by placing test tubes containing equal volumes on opposite sides of the centrifuge. Add one more drop of the 6 M HCl to each solution to test for completeness of precipitations. Centrifuge again if necessary, and decant the supernatant solution from the chloride precipitate. The solution should be saved for further study if ions from other groups may be present and need testing. (For this experiment we are done with the solution and it is now waste.)

2. Separation of Pb^{2+}. Add 1 mL of distilled water to the precipitate in the test tube and place in a 100 mL beaker that is half full of boiling water*. Allow the tube to remain in the boiling water bath for a few minutes and stir occasionally with a glass rod. The higher temperature should start dissolving the $PbCl_2$ solid

   Centrifuge the hot solution and pour it into another test tube and **save the solution for step 3**. **Save the remaining precipitate for testing in step 4**.

   *Note: Watch the level of water in the beaker. Add water when it is low. Never add water to a dry hot beaker as the glass is likely to break.

3. Identification of Pb^{2+}. To the **solution from step 2**, add one drop of 6 M acetic acid and a few drops of 1 M K_2CrO_4. If Pb^{2+} is present in the solution, a yellow precipitate of $PbCrO_4$ will form.

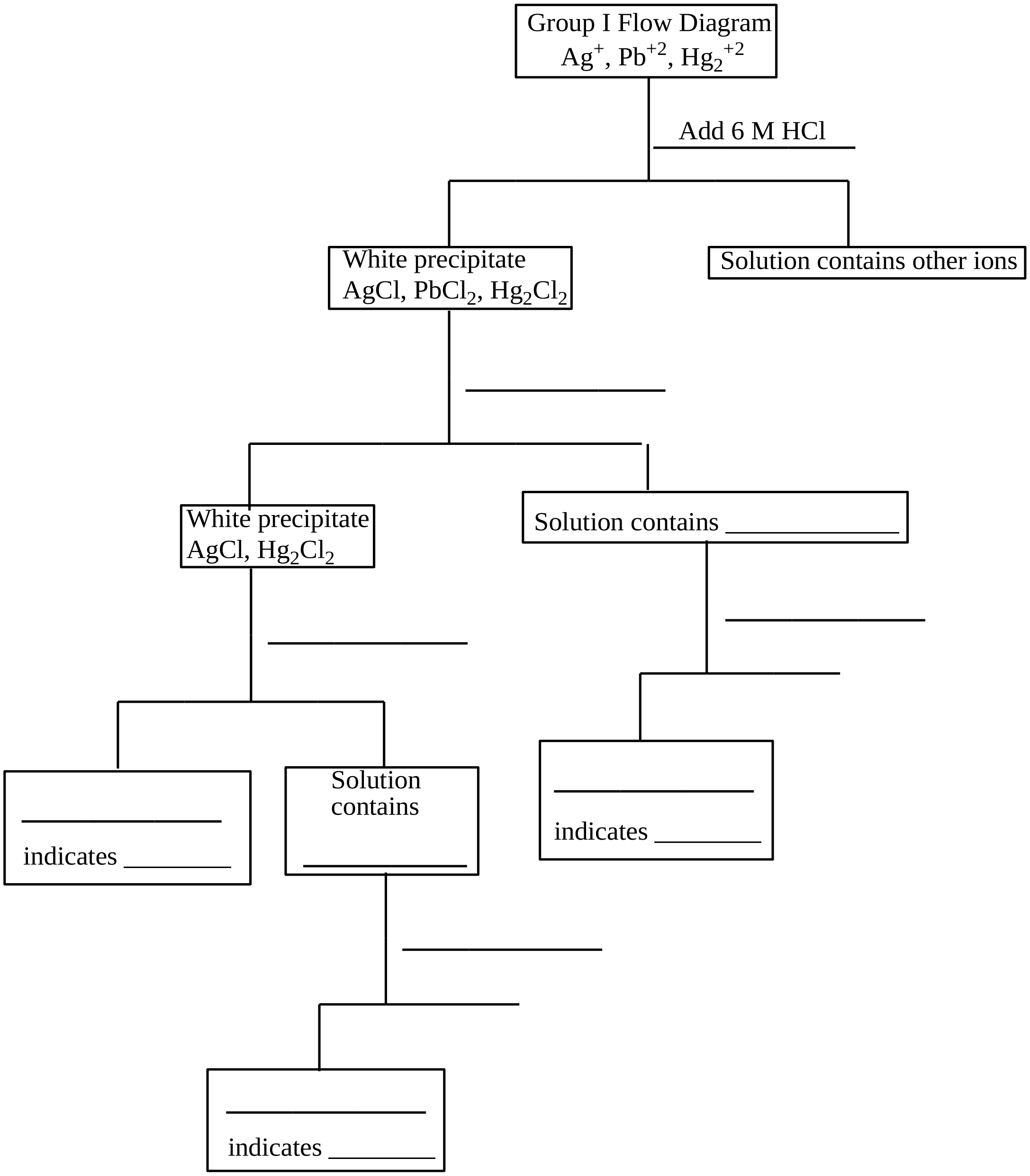
4. Separation and Identification of Hg_2^{2+}. To the **precipitate from step 2**, add 15 to 20 drops of 6 M NH_3 and stir thoroughly. Centrifuge the solution and decant. (**Save the decanted solution for step 5!)** A gray or black precipitate, produced by reaction of Hg_2Cl_2 with ammonia to produce metallic mercury, will establish the presence of Hg_2^{2+}.

5. Identification of Ag^+. To the **solution from step 4**, add 6 M HNO_3 until it is acidic toward litmus paper. (*Remember you previously added 15-20 drops of base).* Test for acidity by dipping the end of your stirring rod in the solution and then touching it to a piece of blue litmus paper (red in acid solution). If Ag^+ is present in the acidified solution, a white precipitate of AgCl will form.

6. If you analyzed ONLY the known solution, then obtain an unknown and analyze it for the possible presence of Ag^+, Pb^{2+}, and Hg_2^{2+}.

Name: ________________________________ Section: ____________________

It is possible to summarize the directions for analysis of the Group I cations in what is called a flow diagram. In the diagram, vertical lines link successive steps in the procedure. Reactant cations or reactant substances containing the ions are at the top end of each line and products formed are at the bottom end. On the product end, a horizontal line separates the <u>solid products on the left</u> and the <u>solution products on the right</u>. Reagents and conditions used to carry out each step are placed alongside the lines. A partially completed flow diagram for the Group I ions follows:



Use this diagram as a brief guide to the procedure. Complete the flow diagram above by directly recording your observations on your known (in the boxes) and unknown (beside the boxes), perhaps using different colored markers.

Name: ___________________________________ Section: ______________________

Experiment Results:

UNKNOWN NUMBER _________ IONS PRESENT _____________________________

Post-Lab Questions: Group I Cations

1. A solution may contain Ag^+, Pb^{2+}, and Hg_2^{2+}. A white precipitate forms on addition of 6 M HCl. The precipitate is partially soluble in hot water. The solid remaining after treatment with hot water turns black on addition of 6 M NH_3. Which of the ions are present, which are absent, and which remain undetermined? State your reasoning below. NOTE: simply listing ions below without the appropriate reasoning will NOT earn you any credit!

Present ____

Absent ____

In Doubt ____

Name: ________________________________ Section: ____________________

Experiment #11 – Qualitative Analysis Scheme of Main Group and Transition Metal Cations[*]

The following experiment is intended to continue the introduction of qualitative analysis through the identification of various main group and transition metal cations. Recall that the analysis of a substance can be extremely difficult given the fact that there are thousands of possibilities for reactions with some similar results. For example, you may have seen from the Group I qualitative analysis that Ag^+, Pb^{+2}, and Hg_2^{+2} all produce a white precipitate when Cl^- is added.

In this experiment, the ions initially chosen were limited to aluminum, the alkaline earth metals, and certain first-row transition metals with the goal of minimizing the amount of hazardous waste generated. Analysis of both a known and unknown solution will test for Al^{+3}, Ba^{+2}, Ca^{+2}, Co^{+2}, Cu^{+2}, Fe^{+3}, Mg^{+2}, Mn^{+2}, and Ni^{+2}. The ions are roughly divided into three groups. The group A ions have very insoluble oxides or hydroxides. The group B ions are poorly coordinated by NH_3 and have very insoluble carbonates; magnesium is included in group B even though it is precipitated separately. Group C metal ions are strongly coordinated by NH_3. This prevents their precipitation in the previous steps.

Note that almost all of the solutions can safely go down the sink in moderate amounts. The barium sulfate should be collected, but its storage and disposal should not pose a serious problem in the small amounts used in the tests. The organic compounds used to test for the transition metal ions should be handled with care as they are irritants and suspected carcinogens. Once in solution, they are used in small amounts that should not pose a serious hazard.

Procedure

Separation of Ion Groups: Unknown and Known (with each group)

1. Place 1 mL of your test solution in a medium test tube.

2. Add l2 drops of 6 M HNO_3, 3 mL of 6 M $NH_3(aq)$, and stir. Wait five minutes, then centrifuge and decant. The solid may contain $Al(OH)_3$, $Fe(OH)_3$, and $Mn(OH)_2$. *Save the solid for Group A analysis*. Use the solution in the next step.

3. To the solution from step 2, add 2 mL of 1 M $(NH_4)_2CO_3$ solution and stir. Centrifuge and decant. The solid may contain $BaCO_3$ and $CaCO_3$. *Save the solid for Group B analysis.* Use the solution in the next step.

---

[*] Adapted from Petty, John T. A Short Qualitative Analysis Scheme without Hazardous Wastes

4. To the solution from step 3, add 1 mL of 1 M Na_2HPO_4 solution. A white precipitate of $MgHPO_4$ indicates the presence of magnesium ion. Centrifuge and decant. *Save the solution for Group C analysis*.

$$Mg^{+2}(aq) + HPO_4^{-2}(aq) \rightarrow MgHPO_4(s) \qquad (1)$$

5. To expedite your time, you should begin Group C step 1 before beginning Group A below as it takes awhile to evaporate the Group C ions to dryness. While waiting for the solution in the evaporating dish as instructed in C-1 to dry, carry out Groups A and B analyses.

Analysis of the Groups of Ions: Group A (Al^{3+}, Fe^{3+}, Mn^{2+})

1. Add 6 M NaOH to solid A from step 2 until pH > 7 (about 1 mL) and stir vigorously with a glass stirring rod. Centrifuge and decant. In strong base, $Al(OH)_3$ solid will dissolve back into the solution, forming the complex ion $Al(OH)_4^-$.

2. To the solution, add 1 M H_3PO_4 dropwise until slightly acidic. A white precipitate indicates aluminum ion.

$$Al(OH)_4^-(aq) + H^+(aq) \rightarrow Al(OH)_3(s) + H_2O(l) \qquad (2)$$

3. To the solid from step 1, add 2 mL of H_2O. Then add 6 M HNO_3 dropwise until acidic and stir to dissolve the solid in order to isolate Fe^{+3} and Mn^{+2} ions. To ensure a low pH, once solution is acidic, add 2 MORE mL of HNO_3 and divide the solution into two test tubes.

4. To one test tube, add 2 – 3 drops of 0.5 M KSCN. A dark-red solution indicates iron(III) ion.

$$Fe^{+3}(aq) + SCN^-(aq) \rightarrow FeSCN^{+2}(aq) \qquad (3)$$

5. To the other test tube, add a spatula tip of sodium bismuthate ($NaBiO_3$). Mix and wait five minutes. A purple solution indicates manganese(II) ion has oxidized to MnO_4^-. (The color of the solution will fade on standing as it reacts in the air.)

$$2Mn^{+2}(aq) + 14H^+(aq) + 5BiO_3^-(aq) \rightarrow \underset{\text{purple}}{2MnO_4^-(aq)} + 5Bi^{+3}(aq) + 7H_2O \qquad (4)$$

Name: ________________________________ Section: ____________________

Group B (Ba^{2+}, Ca^{2+})

<u>NOTE</u>: Magnesium ion is technically part of Group B. However, the test for magnesium ion has already been performed in step 4 of the ion group separation.

1. Add 1 mL of water to solid B from step 3 of the ion group separation, and then slowly add 6 M HCl dropwise. Stir until the solid has dissolved.

2. Add 1 mL of l M Na_2SO_4 and stir. A white precipitate indicates barium ion. Centrifuge and decant.

$$Ba^{+2}(aq) + SO_4^{-2}(aq) \rightarrow BaSO_4(s) \qquad (5)$$

3. To the solution, add 3 drops of 6 M $NH_3(aq)$ and 1 mL of 0.3 M $(NH_4)_2C_2O_4$ solution. Stir and let stand for 1 min. The formation of a white precipitate indicates calcium ion.

$$Ca^{+2}(aq) + C_2O_4^{-2}(aq) \rightarrow CaC_2O_4(s) \qquad (6)$$

Group C (Co^{2+}, Cu^{2+}, Ni^{2+})

1. Pour the solution for Group C analysis containing $Co(NH_3)_6^{+2}$, $Cu(NH_3)_4^{+2}$, and $Ni(NH_3)_6^{+2}$ from step 4 of the ion group separation into an evaporating dish and heat gently over a beaker of boiling water in a fume hood to dryness. *Hint: If the dried residue appears purple, this is an indication of the presence of Co^{2+}. If the residue has a greenish color, this is an indication of the presence of Ni^{2+} while a blue color of the residue is an indication of the presence of Cu^{2+}. You still have to carry out the procedure below to further confirm these "hints."*

2. Dissolve the solid from step 1 in 2 mL of 1 M H_3PO_4. Then add 1 mL of 1 M Na_2HPO_4 and divide it equally into three test tubes.

3. To the first test tube add a spatula tip of KI and stir. A brown solution with a pale tan precipitate indicates copper(II) ion.

$$Cu^{+2}(aq) + 2\,I^{-}(aq) \rightarrow CuI_2(s) \qquad (7)$$

4. To the second test tube add Na_2HPO_4 until basic and then add a few drops of 1% dimethylglyoxime solution (DMG^-). A red/pink precipitate indicates nickel ion.

$$Ni(NH_3)_6^{+2}(aq) + 3DMG^{-}(aq) \rightarrow Ni(DMG)_3^{-}(aq) + 6NH_3(g) \qquad (8)$$

5. To the third test tube, add 2 – 3 drops of 0.5% 1-nitroso-2-naphthol in 95% ethanol solution and stir. A red-brown precipitate indicates cobalt(II) ion.

Name: ______________________________ Section: ___________________

Pre-Lab Assignment: Prepare a complete flow diagram for the separation and identification of the various cations presented in this experiment.

Name: ________________________________ Section: ____________________

Experiment Results:

UNKNOWN______ IONS PRESENT__________________________

Post-Lab Questions:

1. The precipitates of iron(III) ion and manganese(II) ion are hydroxides. What is the purpose of adding 6 M $HNO_3(aq)$ in step 3 of the Group A analysis?

2. In step 2 of Group B, Na_2SO_4 is added. A white precipitate indicates the presence of barium ion. Look up and write down the appropriate K_{sp} values and suggest a reason why a calcium precipitate does not form along with the barium precipitate.

3. Describe any modifications (if any) that you discovered that worked for your analysis. How would you improve this experiment?

Name: ________________________________ Section: ____________________

Experiment #12 – Qualitative Analysis of Common Anions

The principles that are employed in the identification of cations can also be applied to the analysis of anions. The qualitative detection of anions in a sample depends on the distinctive solubility properties of particular salts of the ions and specific chemical reactions that are (ideally) unique to a particular ion. In this experiment, we will explore ways to detect the presence of CO_3^{-2}, SO_4^{-2}, PO_4^{-3}, Cl^-, and I^-. You will be testing both known and unknown solutions.

Procedure

1. Test for the Sulfate Ion

   To 1 mL of the test solution, add 6 M HNO_3 drop by drop until the solution is acidic. Then add 1 mL of 0.1 M $BaCl_2$ solution in order to produce a white precipitate of $BaSO_4$.

$$SO_4^{-2}(aq) + Ba^{+2}(aq) \rightarrow BaSO_4(s) \qquad (1)$$

2. Test for the Carbonate Ion

   To 1 ml of a new test solution, observe to see if any gas bubbles form while adding 20 drops of 6 M HNO_3. Verify it is acidic. This gas formation is a strong indication of the presence of CO_3^{2-}.

$$CO_3^{2-}(aq) + 2H^+(aq) \rightarrow CO_2(g) + H_2O(l) \qquad (2a)$$

3. Test for the Chloride Ion

   To 1 mL of a new test solution, add a couple drops of 6 M HNO_3 as needed to make the solution slightly acidic. Add 10 drops of 0.1 M $AgNO_3$. No precipitate proves the absence of Cl^-, Br^-, or I^-. Centrifuge the mixture. Test the clear filtrate with 1 drop of 0.1 M $AgNO_3$ for complete precipitation. If necessary, centrifuge again. Discard the filtrate. To this precipitate, add 1 mL of D.I. water, 2 drops of 6 M NH_3, and 6 drops of 0.1 M $AgNO_3$. The proportions are important, since we want to dissolve ONLY AgCl.

$$AgCl(s) + 2NH_3(aq) \rightarrow Ag(NH_3)_2^+(aq) + Cl^-(aq) \qquad (3a)$$

   Shake the mixture well and centrifuge. Transfer the clear solution to a clean test tube, and acidify once again with 6 M HNO_3. A white precipitate of AgCl confirms the presence of Cl^-.

$$Ag(NH_3)_2^+(aq) + Cl^-(aq) + 2H^+ \rightarrow AgCl(s) + 2NH_4^+(aq) \qquad (3b)$$

Name: ___________________________________ Section: ______________________

4. Test for the Iodide Ion

   Acidify a 2 mL sample of a new test solution by adding 6 M HCl. Add 1 mL of 0.1 M $FeCl_3$ to oxidize any I^- to I_2. Add 1 mL of hexane and agitate the mixture. A purple color of I_2 in the hexane layer indicates I^- was present in the original sample.

$$2\ I^-(aq) + 2\ Fe^{+3}(aq) \rightarrow I_2(aq) + 2Fe^{+2}(aq) \qquad (4)$$

5. Test for the Phosphate Ion

   (A) If no I^- was present, mix about 2 drops of 0.5 M $(NH_4)_6Mo_7O_{24}$ reagent with 5 drops of 6 M HNO_3 to 1 mL of a new test solution.

   (B) If I^- was present, add 5 drops of 6 M HNO_3 to 1 mL of a new test solution and boil the test tube for 5 to 10 minutes to remove the iodide. Then add 2 drops of the ammonium molybdate reagent to the test solution.

   A yellow precipitate of ammonium phosphomolybdate, $(NH_4)_3PO_4{\cdot}12MoO_3$, appearing at once or after the mixture has been warmed a few minutes to 40 °C indicates the presence of PO_4^{-3}.

$$21NH_4^+(aq) + 12[Mo_7O_{24}^{-6}](aq) + 7H_3PO_4(aq) + 51H^+(aq) \rightarrow$$
$$7(NH_4)_3PO_4 \cdot 12MoO_3(aq) + 36\ H_2O(l) \qquad (5)$$

Record your observations for your known and unknown solutions below. Determine the identity of your unknown.

Experiment Observations and Results:

UNKNOWN______ IONS PRESENT____________________________

Name: ________________________________ Section: ____________________

Pre-Lab Assignment:

1. Construct separate flow charts for the identification of the various five anions in a known sample. Refer to Experiment #10 for guidelines on preparing your flow charts.

Name: ________________________________ Section: ____________________

Post-Lab Assignment: Anion Analysis

1. A solution may contain Cl^{-}, CO_3^{-2}, PO_4^{-3}, and/or SO_4^{-2}. No effect is observed upon addition of 6 M HNO_3; this resulting mixture will be referred to as solution 1. No effect is observed on addition of 0.1 M $AgNO_3$ to solution 1. A white precipitate is reported on addition of 1 M $BaCl_2$ to solution 1. Finally, a yellow precipitate is observed on addition of 0.5 M $(NH_4)_6Mo_7O_{24}$ to solution 1. Which of the ions are present, which are absent, and which remain undetermined? State your reasoning below. NOTE: simply listing ions below without the appropriate reasoning will NOT earn you any credit!

Present ____

Absent ____

In Doubt ______________________________________

Name: ________________________________ Section: ____________________

Experiment #13 – Electrochemical Cells[*]

If a copper strip is placed in a solution of copper ions, one of the following reactions may occur:

$$Cu^{2+} + 2e^{-} \rightarrow Cu$$
$$Cu \rightarrow Cu^{2+} + 2e^{-}$$

The electrical potential that would be developed by these reactions prevents their continuation. These reactions are called half-reactions or half-cell reactions. There is no direct way to measure the electrical potential (electromotive force, emf) of a half-cell reaction. Similarly, a zinc strip in a solution of zinc ions has the possible reactions:

$$Zn^{2+} + 2e^{-} \rightarrow Zn$$
$$Zn \rightarrow Zn^{2+} + 2e^{-}$$

But these are also prevented from occurring by the electrical potential that would build up. If the metal electrodes (copper and zinc) in the two solutions are connected by a wire, and if the solutions are electrically connected by perhaps a porous membrane or a bridge that minimizes mixing of the solutions, a flow of electrons will move from one electrode, where the reaction is $M_1 \rightarrow M_1^{n+} + ne^{-}$, to the other electrode, where the reaction is $M_2^{n+} + ne^{-} \rightarrow M_2$.

In this case, the zinc metal goes into solution as zinc ions, and the copper ions plate out. The overall cell reaction is:

$$Zn + Cu^{2+} \rightarrow Zn^{2+} + Cu$$

The electromotive force for such a cell, which is written as $Zn|Zn^{+2}||Cu^{+2}|Cu$, can be measured.

By convention, all half-cell emf's are compared to the emf of the standard hydrogen electrode. The standard hydrogen electrode is defined as a platinum electrode covered with platinum black that is in contact with hydrogen gas at 1 atmosphere pressure and a 1 molar solution of hydronium ions (actually, it is defined for unit activity). The hydrogen electrode half-cell reaction is

$$2H^{+} + 2e^{-} \rightarrow H_2$$

The emf of a half-cell, with respect to the standard hydrogen electrode, is called the *reduction potential.* Standard reduction potentials, $\boldsymbol{E}^{o}$, are for 1-molar solutions.

---

[*] adapted from C. B. Anderson, Basic Experimental Chemistry

Consequently, the difference between the oxidation potentials of two half-cells is the emf they would develop if connected together as a cell. The emf for the Zn-Cu cell described would be:

$$\begin{array}{cccccc} \boldsymbol{E}^\circ_{ox}\,(Zn \rightarrow Zn^{2+}) & + & \boldsymbol{E}^\circ_{red}\,(Cu^{2+} \rightarrow Cu) & = & \boldsymbol{E}^\circ_{cell} \\ -(-0.76\text{ V}) & + & +0.34\text{ V} & = & 1.10\text{ V} \end{array}$$

If a cell reaction can be written as

$$a\mathrm{A} + b\mathrm{B} \rightarrow cC + d\mathrm{D}$$

then the emf of the cell can be expressed in the form of the following equation, developed by Nernst:

$$\boldsymbol{E} = \boldsymbol{E}^\circ - (\mathrm{RT} / n\mathbf{F}) \ln ([\mathrm{C}]^c [\mathrm{D}]^d / [\mathrm{A}]^a [\mathrm{B}]^b)$$

If all the concentrations are 1 M, then the natural logarithmic term becomes zero and $\boldsymbol{E} = \boldsymbol{E}^o$, which is the reason for choosing 1-molar concentration as the standard condition. If the system is at equilibrium, then

$$\frac{[\mathrm{C}]^c[\mathrm{D}]^d}{[\mathrm{A}]^a[\mathrm{B}]^b}$$

and the emf developed by such a cell at equilibrium must be zero. Therefore,

$$0 = \boldsymbol{E}^\circ - (\mathrm{RT} / n\mathbf{F}) \ln \mathbf{K}_c \quad \text{-OR-} \quad \ln \mathbf{K}_c = n\mathbf{F}\boldsymbol{E}^\circ / \mathrm{RT}$$

In these equations, **F** is Faraday's Constant (96,485 C / mol e^-), *n* is the number of electrons transferred in the oxidation-reduction step, and R is the gas constant in units of electrical work (8.314 J / K mol).

To measure the emf of a galvanic cell, a sensitive meter is needed, but it is important that the meter not draw a significant amount of current. If the current produced by the cell to be measured is large, the cell will become polarized, and the emf will be decreased. Many solid state voltmeters have sufficiently high impedance that they can be used to measure the emf of a cell accurately.

Procedure

Part 1: The Daniell Cell

Part 1A: Standard Conditions Place 1.0 M $ZnSO_4$ solution in a 25-mL beaker and 1.0 M $CuSO_4$ solution in another beaker of the same size. The liquid levels should be the same. In the zinc solution, place a clean strip of zinc, and in the copper solution, a clean strip of copper. The zinc strip may be cleaned by dipping it in a beaker of about 2 M HCl. The copper strip can be cleaned by dipping it in a beaker of 2 M HNO_3. Rinse the electrodes with deionized water thoroughly before using them.

Name: ______________________________ Section: ____________________

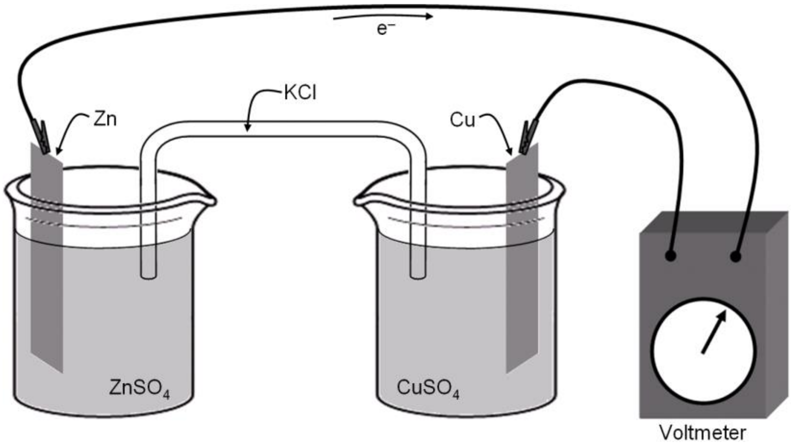


Figure 1

Obtain a multimeter along with one red and one black wire. Set up the meter to measure 2V DC Voltage. Attach the red wire to the V port and the black wire to the COM port. Note the sign of the voltage; it must be positive (+) or reverse which electrode the red and black probes are touching. When $E°_{cell}$ is positive, the black electrode is the anode. The cells are connected with a cotton length soaked in 0.5 M potassium chloride (see Figure 1 above). Electrons will flow from the zinc electrode to the copper electrode because of the reactions occurring in the half-cells:

$$Zn \rightarrow Zn^{2+} + 2e^-$$
$$Cu^{2+} + 2e^- \rightarrow Cu$$

The overall reaction for the cell is:

$$Cu^{2+} + Zn \rightarrow Cu + Zn^{2+}$$

Measure the voltage of this cell.

Next measure the voltage of a couple of non-standard Daniell cells following the same procedure:
Part 1B: 0.10 M ZnSO4 and 1.0 M CuSO4
Part 1C: 1.0 M ZnSO4 and 0.10 M CuSO4

Part 2: Solubility Product of Copper(II) Hydroxide
If an electrochemical cell can be built in which the half-cell reactions are:

$$Cu^{2+} + 2e^- \rightarrow Cu$$
$$Cu + 2\,OH^- \rightarrow Cu(OH)_2(s) + 2e^-$$

the overall cell reaction is the reverse of the solubility product of cupric hydroxide:

$$Cu^{2+} + 2OH^- \rightleftharpoons Cu(OH)_2(s)$$

From the Nernst equation, we know that:

$$\boldsymbol{E} \;=\; \boldsymbol{E}° - \frac{\mathrm{RT}}{2\mathbf{F}} \ln \frac{1}{[\mathrm{Cu}^{2+}][\mathrm{OH}^{-}]^{2}}$$

The inverse natural logarithm changes the equation to:

$$\boldsymbol{E} \;=\; \boldsymbol{E}° + \frac{\mathrm{RT}}{2\mathbf{F}} \ln\, [\mathrm{Cu}^{2+}][\mathrm{OH}^{-}]^{2}$$

Solving for the standard reduction potential results in:

$$\boldsymbol{E}° \;=\; \boldsymbol{E} - \frac{\mathrm{RT}}{2\mathbf{F}} \ln\, [\mathrm{Cu}^{2+}][\mathrm{OH}^{-}]^{2}$$

Consequently, if we can measure the emf, $\boldsymbol{E}$, of such a cell, we can calculate $\boldsymbol{E}°$ if we know the concentration of cupric ion in one half-cell and the concentration of hydroxide ion in the other half-cell.

In the introductory discussion, we have shown from the Nernst equation that the following relationship exists:

$$\ln \mathbf{K}_c \;=\; n\mathbf{F}\boldsymbol{E}°/\mathrm{RT}$$

Therefore, if $\boldsymbol{E}°$ is experimentally obtained, it can be converted into a value of the solubility product $\mathbf{K}_{sp}$. Also, if we use a literature value for $\mathbf{K}_{sp}$, we can calculate the theoretical $\boldsymbol{E}°$.

Construct a cell of two beakers. In a 25-mL beaker, place 1.0 M $CuSO_4$ solution and a clean copper strip. The copper strip can be cleaned in dilute nitric acid, but it must be washed well with distilled water. Until it is put into the copper sulfate solution, it should be kept in dilute HNO_3 but rinsed with deionized water before use. In another 25-mL clean beaker, add the same height of 1.0 M KOH solution plus 10 drops of 1.0 M $CuSO_4$ solution. Most of the copper ions will form $Cu(OH)_2$ (s) in equilibrium with the 1.0 M solution of hydroxide ions. Add a copper electrode to this KOH solution. Connect wires to the electrodes and the appropriate terminals of the voltmeter or potentiometer. The KOH side is the negative electrode, because copper goes into the oxidized form (Cu^{2+}) because of the low concentration of Cu^{2+} in the KOH solution. Now add a new salt bridge made of a cotton length soaked in 0.5 M KCl. Measure the emf of the cell as soon as possible. The cell is polarized very easily. To get around this difficulty, clean the electrodes again, rinse, and return them to the solutions. Re-measure the voltage. Calculate the solubility product, K_{sp}, using the equations above. Literature values range from 10^{-14} to 10^{-20}, the latter being considered the best value. Taking the standard $\boldsymbol{E}°$ of the Cu-Cu^{2+} half-cell to be –0.34 V, evaluate $\boldsymbol{E}°$ for the Cu-$Cu(OH)_2$, OH^- half-cell.

Name: ________________________________ Section: ____________________

Part 3: Formation Constant of Tetraamminecopper(II) cation

If we construct a cell having the half-reactions:

$$Cu^{2+} + 2e^- \rightarrow Cu$$

$$Cu + 4NH_3 \rightarrow Cu(NH_3)_4^{2+} + 2e^- \quad \text{(Look up the cell potential!)}$$

the overall cell reaction is the formation constant of the tetraamminecopper(II) cation:

$$Cu^{2+} + 4NH_3 \rightleftharpoons Cu(NH_3)_4^{2+}$$

Just like before, the copper strip can be cleaned in dilute nitric acid, but it must be washed well with distilled water. Until it is put into the copper sulfate solution, it should be kept in dilute HNO_3 but rinsed with deionized water before use. In a 25-mL beaker, place 1.0 M aqueous ammonia, 10 drops of 1.0 M $CuSO_4$ solution, and a clean copper strip. In another 25-mL beaker, add the same height of 1.0 M $CuSO_4$ solution and another rinsed copper electrode. Connect the electrodes to the voltmeter. Make the electrical connection by adding a new salt bridge made of a cotton length soaked in 0.5 M KCl. Determine the voltage of the cell, and then calculate K_f.

Name: ______________________________ Section: ___________________

Pre-Lab Assignment: Electrochemical Cells Experiment

Answer each of the following questions and place the responses on the lines provided.

1. The following data were measured using a nickel electrode as the standard:

$Cu^{2+}(aq) + 2e^{-} \rightarrow Cu(s)$	0.62 V
$Ni^{2+}(aq) + 2e^{-} \rightarrow Ni(s)$	0.00 V
$Fe^{2+}(aq) + 2e^{-} \rightarrow Fe(s)$	–0.15 V
$Al^{3+}(aq) + 3e^{-} \rightarrow Al(s)$	–1.38 V

A. Which ion is most easily reduced?

____

B. Which metal is most easily oxidized?

____

C. The copper and aluminum electrodes are connected in a battery.

1) Which is the anode? cathode?

Anode: ________________ Cathode: ________________

2) Which is oxidized? reduced?

Oxidized: ________________ Reduced: ________________

3) What will the battery voltage be?

____

4) Write a balanced net ionic equation for the reaction that takes place.

____

Name: ___________________________________ Section: ______________________

Experiment

Part 1A**:** E^{o} for: Zn | 1.0 M Zn^{+2} || 1.0 M Cu^{+2} | Cu

Theoretical: __________ V Experimental: __________ V

Part 1B**:** E_{cell} for: Zn | 0.10 M Zn^{+2} || 1.0 M Cu^{+2} | Cu

Theoretical: __________ V Experimental: __________ V

Part 1C**:** E_{cell} for: Zn | 1.0 M Zn^{+2} || 0.10 M Cu^{+2} | Cu

Theoretical: __________ V Experimental: __________ V

Name: ___________________________________ Section: ______________________

Part 2: E^o and K_{sp} for: $Cu|Cu(OH)_2$ in 1.0 M OH^- || 1.0 M Cu^{2+} | Cu

Experimental E^o = __________ V

Calculate experimental **K**sp using experimental E^o and the Nernst equation at equilibrium.

Experimental K_{sp} = __________

Calculate the theoretical E^o using a literature value for **K**sp = 2.2×10^{-20}.

Theoretical E^o = __________ V

Calculate the % error in E^o.

% error in E^o = __________

Name: ___________________________________ Section: ______________________

Part 3: $\boldsymbol{E}^{o}$ and $\mathbf{K_f}$ for: $Cu|Cu(NH_3)_4^{2+}$ in 1.0 M NH_3 || 1.0 M Cu^{2+} | Cu

Experimental $\boldsymbol{E^o}$ **=** __________ V

Calculate experimental $\mathbf{K_f}$ using experimental $\boldsymbol{E}^{o}$ and the Nernst equation at equilibrium.

Experimental K_f = __________

Calculate the theoretical $\boldsymbol{E}^{o}$ using a literature value for $\mathbf{K_f} = 1.7 \times 10^{13}$.

Theoretical $\boldsymbol{E^o}$ **=** __________ V

Calculate the % error in $\boldsymbol{E}^{o}$.

% error in $\boldsymbol{E}^{o}$ = __________

Name: ______________________________ Section: ___________________

Post-Lab Questions:

1. Sketch and label well with movement arrows the processes occurring in the standard Daniell cell (from Part 1A) while it is operating. Note that the electrode reactions produce and consume ions at the electrodes.

2. Briefly comment on how well your experimental cell potentials match the theoretical cell potentials in the standard and nonstandard Daniell cells from parts 1A/1B/1C.

3. How should the voltage change in the Daniell cell if the zinc solution is made more dilute (and the concentration of the copper solution is kept the same)? Explain your choice.

4. Calculate the solubility product of silver chloride, an extremely insoluble salt, from the following oxidation potentials:

$Ag \rightarrow Ag^+ + e^-$ $E^o = -0.7996$ V
$Ag + Cl^- \rightarrow AgCl + e^-$ $E^o = -0.2221$ V

5. Construct a standard voltaic cell diagram that will enable you to determine the solubility product of AgCl from the half reactions given in the previous question.

6. A cell that has the following half reactions was constructed.

$Cd^{2+} + 2e^- \rightarrow Cd$
$Cd + 3\text{ en} \rightarrow Cd(en)_3^{2+} + 2e^-$

The measured E^o at 25 °C was +0.38 V. Calculate the experimental K_f.

Name: ________________________________ Section: ____________________

Experiment #14 – Determination of Equivalent Mass by Electrolysis

The equivalent mass of an element can be related to the chemical effects observed in electrolysis. Because they can contain ions, some liquids will conduct an electric current. If the two terminals on a storage battery or any other source of DC voltage are connected through metal electrodes to a conducting liquid, an electric current will pass through the liquid and chemical reactions will occur at the two metal electrodes. In this process, electrolysis is said to occur, and the liquid is said to be electrolyzed.

At the electrode connected to the negative pole or cathode of the battery, a reduction reaction will invariably be observed. In this reaction, electrons will be accepted by one of the species present in the aqueous solution. The species reduced will ordinarily be a metallic cation, the H^+ ion, or possibly water itself. The reaction that is observed is the one that occurs with the least expenditure of electrical energy and will depend on the composition of the solution and overpotentials that may be involved. For this experiment, hydrogen gas is produced by the reduction of the hydrogen ion:

$$2H^+(aq) + 2e^- \rightarrow H_2(g) \qquad (1)$$

At the positive pole of an electrolysis cell (i.e. the metal electrode that is connected to the positive terminal of the battery), an oxidation reaction will occur at the anode, in which some species give up electrons. Again, this may involve an ionic or neutral species in the solution or the metallic electrode itself. For this experiment, the pertinent oxidation reaction for an unknown metal occurs as follows:

$$M(s) \rightarrow M^{n+}(aq) + ne^- \qquad (2)$$

During the course of the electrolysis, the atoms in the metal electrode will be converted to metallic cations and will go into the solution. The mass of the metal electrode will decrease, depending on the amount of electricity passing through the cell and the nature of the metal. To oxidize one mole or one molar mass of the metal, it would take n faradays, where n is the charge on the cation that is formed. By definition, one faraday of electricity would cause one gram equivalent mass, GEM, of metal to go into solution. The molar mass, MM, and the equivalent mass of the metal are related by the equation:

$$\text{MM} = \text{GEM} \times n \qquad (3)$$

In an electrolysis experiment, since n is not determined independently, it is not possible to find the molar mass of a metal. It is possible, however, to find gram equivalent masses of many metals.

In this experiment, a sample of an unknown metal is oxidized at the positive pole (anode) of an electrolysis cell, weighing the metal before and after the electrolysis, thereby determining its loss in mass. Using the same amount of electricity and number of electrons, the hydrogen ion is reduced at the negative pole of the electrolysis cell. From the volume of H_2 gas that is produced under known conditions, we can calculate the number of moles of H_2 formed and hence the number of faradays that passed through the cell. The equivalent mass of the metal is then calculated as the amount of metal that would be oxidized if one faraday were used. In the last part of the experiment, your instructor will tell you which metal you used. Using equation 3, it will be possible to determine the charge on the metallic cations that was produced during electrolysis.

Procedure

Obtain a buret and sample of unknown metal. Lightly sand the metal to clean it. Rinse the metal with water and then in acetone. Let the acetone evaporate. When the sample is dry, weigh the unknown metal on the analytical balance to 0.001 g.

Set up the electrolysis apparatus as indicated in Figure One (next page). Add about 100 mL of a pre-mixed solution of 0.5 M $HC_2H_3O_2$ in 0.5 M Na_2SO_4 in the beaker with the gas buret. This will serve as the conducting solution. Insert the bare coiled end of the heavy copper wire up into the end of the buret; all but the coiled end of the wire should be covered with watertight insulation. Immerse the end of the buret in the solution and attach a length of rubber tubing to its upper end. SLOWLY AND CAREFULLY open the stopcock on the buret and, with vacuum suction, carefully draw the acid up to the top of the graduations. Close the stopcock. Check the solution level after a few minutes to make sure the stopcock does not leak. Record the level.

The unknown metal will serve as the anode in the electrolysis cell. Connect the metal to the positive pole of the power source with an alligator clip and immerse the metal but not the clip in the conducting solution. Note: The alligator clip must connect to the metal and not the non-conducting tape that indicates the unknown number of the metal. The bare coiled wire inside the buret will be the cathode in the cell. Connect the cathode to the negative pole of the power source. Hydrogen gas should immediately begin to bubble from the cathode. Collect the gas until about 30 mL have been produced. At that point, stop the electrolysis by disconnecting the copper electrode from the power source. Record the level of the liquid in the buret. Measure and record the temperature and the barometric pressure in the laboratory. Note that in some cases, cloudiness may develop in the solution during the electrolysis; this is caused by the formation of a metal hydroxide and will have no adverse effect on the experiment.

Raise the buret and discard the conducting solution from the beaker into the labeled waste container. Rinse the beaker with water, and pour in 100 mL of fresh conducting solution. Take the alligator clip off the metal anode and wash the anode with 0.1 M $HC_2H_3O_2$. Rub off any loose adhering coating with your fingers, and then rinse off your hands. Rinse the electrode in water and then in acetone. Let the acetone evaporate. Weigh the dry metal electrode to the nearest 0.001 g.

Name: ________________________________ Section: ____________________

Reassemble the apparatus and repeat the electrolysis, once again generating about 30 mL of H_2 and recording the initial and final liquid levels in the buret. Perform the necessary calculations to determine the charge of your unknown cation.

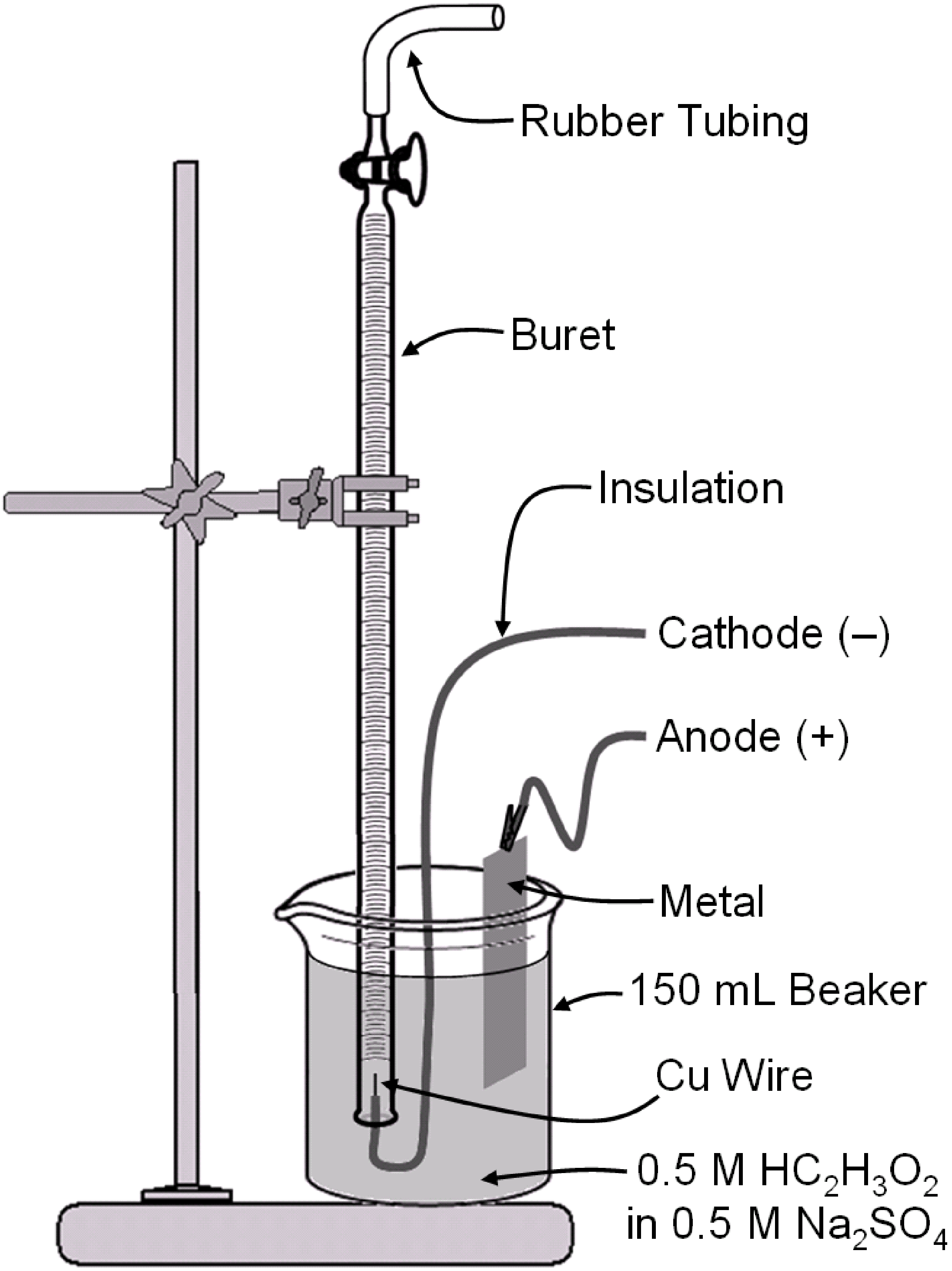


Figure 1

Name: ________________________________ Section: ____________________

Pre-Lab Questions: Determination of an Equivalent Mass by Electrolysis

1. In an electrolysis cell similar to the one employed in this experiment, a student observed that his unknown metal anode lost 0.233 g while a total volume of 94.50 mL of H_2 was being produced. The temperature in the laboratory was 25 ºC, and the barometric pressure was 740 mm Hg. At 25 ºC, the vapor pressure of water is 23.8 mm Hg. To find the equivalent mass of his metal, the student filled in the blanks below:

   $P_{\text{hydrogen gas}} = P_{\text{bar}} - VP_{\text{water}}$ = ________________ mm Hg = ________________ atm

   $V_{\text{hydrogen gas}}$ = ________________ mL = ________________ L

   T = ________________ K

   $n_{\text{hydrogen gas}}$ = ________________ moles

   1 mol H_2 requires passage of ________________ faradays

   Faradays passed (moles of electrons) = ________________

   Loss of mass of metal anode = ________________ g

   Grams of metal lost per faraday passed = grams lost/faradays passed =

   ________________ g = GEM

   The student was told that the identity of the metal anode is copper.

   MM Cu = ______________ g. The charge *n* on the Cu ion is ______________. (Eq. 3)

Name: ______________________________ Section: ___________________

Data and Calculations: Determination of an Equivalent Mass by Electrolysis

Mass of metal anode ____________________ g

Mass of anode after first electrolysis ____________________g

Mass of anode after second electrolysis ____________________ g

Initial buret reading __________________ mL

Buret reading after first electrolysis __________________ mL

Initial buret reading for the second electrolysis __________________ mL

Buret reading after second electrolysis __________________ mL

Barometric pressure ________________ mmHg

Temperature *T* ___________________ °C

Vapor pressure of H_2O at *T* ________________ mmHg

Total volume of H_2 produced, *V* __________________ mL

Temperature *T* ___________________ K

Pressure exerted by dry H_2: $P = P_{bar} - VP_{water}$
(ignore any pressure effect due to liquid levels in the buret) ________________ mmHg

Moles of H_2 produced, *n* _________________ moles

Faradays passed (moles of electrons) _____________________

Loss in mass by anode ____________________ g

Equivalent mass of metal (GEM = g lost / faradays passed) ____________________ g

Unknown metal number _____________________

Metal _____________________

MM ____________________ g

Charge *n* on cation (equation 3) _____________________

Name: ______________________________ Section: ____________________

Pre-Lab Questions: Determination of an Equivalent Mass by Electrolysis

1. In ordinary units, the faraday is equal to 96,485 coulombs. A coulomb is the amount of electricity passed when a current of one ampere flows for one second. Given the charge on an electron, 1.6022 x 10^{-19} coulombs, calculate a value for Avogadro's number.

____

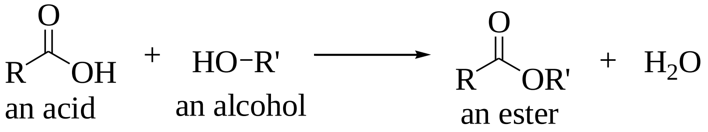
2. Consider the electrolysis of Na_2SO_4(aq). Write the overall net ionic equation that occurs for this electrolysis. Note: Consider all the possible reactions; the oxidation of water or sulfate ions, the reduction of water or sodium ions. Determine which is more likely to occur.

____

Name: ______________________________ Section: ____________________

Experiment #15 – Synthesis and Characterization of Aspirin

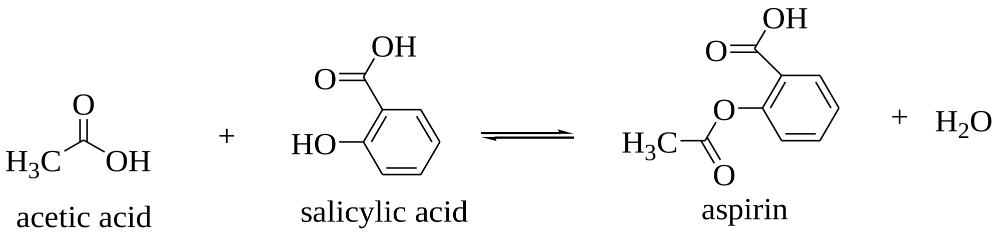
One of the simpler organic reactions that can be carried out is the formation of an ester from an acid and an alcohol. This reaction proceeds as follows:



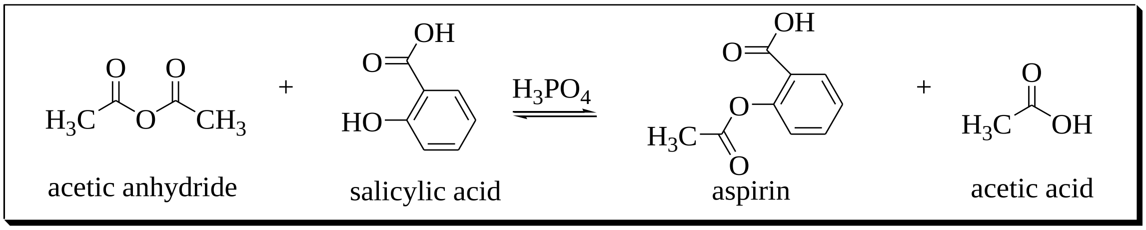
In the equation, R and R’ are H atoms or organic fragments like CH_3, C_2H_5, or more complex aromatic groups. There are many known esters in organic chemistry that can be synthesized from organic acids and alcohols. The driving force for the reaction is in general not very great, resulting in an equilibrium mixture of the formed ester, water, acid, and alcohol.

There are some esters which are solids because of their high molecular weight or other properties. Most of these esters are not soluble in water, so they can be separated from the mixture by crystallization. This experiment involves an ester of this type, a substance commonly called aspirin (or acetylsalicylic acid). Aspirin is the active component in headache pills and is one of the most effective, relatively nontoxic, pain killers.

Aspirin can be made by the reaction of the hydroxyl group (–OH group) in the salicylic acid molecule with the carboxylic acid group (–COOH group) in acetic acid. The reaction proceeds as follows:



A better preparative method, which we will use in this experiment, employs acetic anhydride in the reaction instead of acetic acid. The anhydride can be considered to be the product of a reaction in which two acetic acid molecules combine, with the elimination of a molecule of water. The anhydride will react with the water produced in the esterification reaction and will tend to drive the reaction to the right. A catalyst, normally sulfuric or phosphoric acid, is also used to speed up the reaction. The reaction occurs as follows:



The aspirin you will prepare in this experiment is somewhat impure and should certainly not be taken internally, even if the experiment gives you a bad headache. We will attempt to purify the aspirin via recrystallization with ethanol. The purified compound will be characterized by its melting point and by infrared spectroscopy. For further theory on IR spectroscopy, see the "Introduction to IR and NMR Spectroscopy" lab located at the end of this manual.

Safety

During the synthesis steps of the procedure, wear gloves and safety goggles. Both phosphoric acid and acetic anhydride are corrosive and will cause burns if spilled on your skin. Salicylic acid is a skin irritant.

Procedure

Fill a 250 mL beaker approximately 1/4 full of water. Place it on a hot plate and heat to 80 ºC. Watch the temperature, the water should not boil.

Weigh out approximately 500 mg salicylic acid in a 25 mL Erlenmeyer flask. *Perform the next operation in the fume hood*: pipet 1.0 mL of acetic anhydride into the flask in such a way as to wash any crystals of salicylic acid on the walls down to the bottom. Add 5 drops of 85% phosphoric acid to the mixture to serve as a catalyst.

Clamp the flask so that it is immersed in the hot water bath for 10 minutes, stirring the liquid in the flask occasionally with a stirring rod. Once the reaction is complete, remove the flask from the water bath, and CAUTIOUSLY add 10 – 20 drops of water to the mixture to destroy any excess acetic anhydride. There will be some hot acetic acid vapor evolved as a result of the decomposition of any unreacted acetic anhydride.

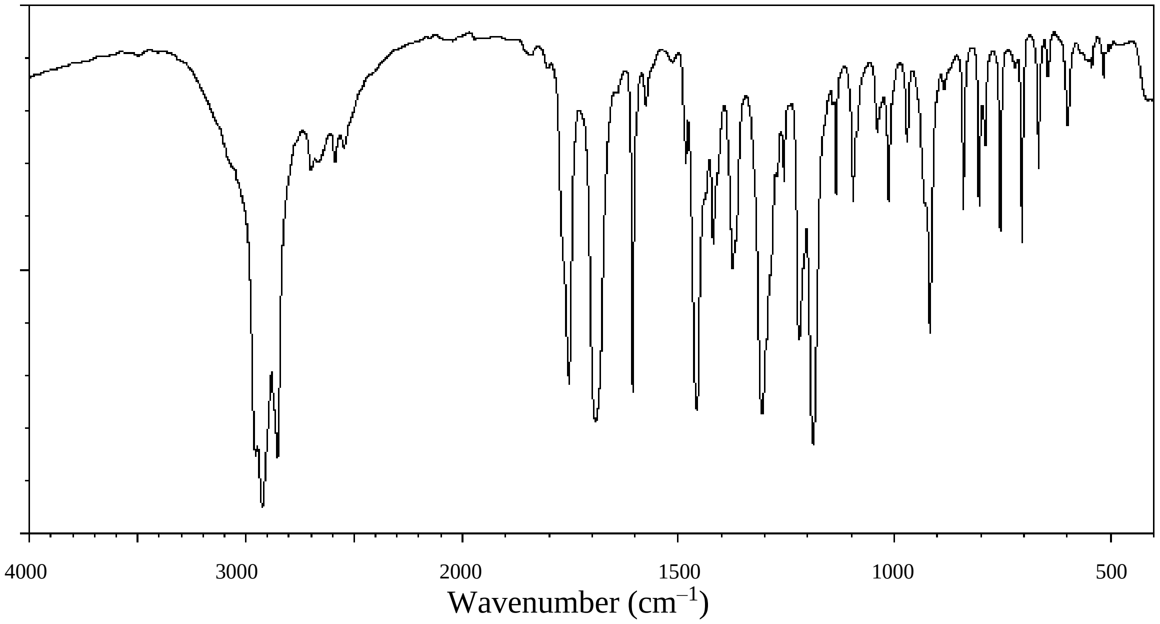
Let the flask cool for a few minutes in air, during which time crystals of aspirin should begin to form. Put the flask in an ice bath to hasten crystallization and increase the yield of product. If crystals are slow to appear, it may be helpful to scratch the inside of the flask with a glass rod. Collect the aspirin by vacuum filtration. Be sure to wet the filter paper and turn on the vacuum suction before transferring the crystals. Use minimal water. Drop distilled water over the crystals; repeat the washing process, and then draw air through the funnel for a few minutes to help dry the crystals. Determine the mass of your impure aspirin.

To purify your synthesized aspirin, transfer it to a 10 mL beaker and add approximately 10 to 20 drops of ethyl alcohol using a plastic pipet. Warm the solution to 60 ºC. Cover the solution and allow it to cool undisturbed to room temperature. Then set the beaker in an ice bath and once again scratch the inside of the flask with a glass rod to induce recrystallization. Collect the purified aspirin by vacuum filtration, and let the crystals dry for a few minutes before weighing them. Determine the mass of your dry purified aspirin.

Record the temperature of the melting point range temperature of your synthesized compound using the melting point apparatus. This range extends from the temperature when the solid begins to soften until the disappearance of the last solid. Make sure your sample is dry before you take this measurement. Place a small amount of your compound in a capillary tube. Place the sample in the melting point apparatus and heat rapidly until 120 °C and then slowly (2 °C / minute) until the sample melts. Find the published value for the melting point of aspirin, and compare it to your obtained experimental value.

Optional, you may be asked to run an IR spectrum of your recrystallized product, where it will be compared to a known spectrum available below. Under the supervision of your instructor, you will take a small sample of your dry purified aspirin and analyze via a Fourier Transform Infrared (FTIR) spectrophotometer. Functional group recognition amongst the various absorption peaks should be noted. Consider the following:

Figure One. IR Spectrum of Acetylsalicylic Acid



C=C (aromatic), 1600 – 1400 cm^{-1}
C=O (ester), 1750 – 1730 cm^{-1}
C=O (carboxylic acid), 1725 – 1700 cm^{-1}
C–O (ester/carboxylic acid), 1300 – 1000 cm^{-1}
O–H (carboxylic acids), 3300 – 2500 cm^{-1}

Name: ______________________________ Section: ____________________

Data

Weight of salicylic acid added ________________________________

Volume of acetic anhydride ________________________________

Density of acetic anhydride ________________________________

Molecular Weight of acetic anhydride ________________________________

Molecular Weight of salicylic acid ________________________________

Theoretical Yield of aspirin ________________________________

Actual Yield of crude aspirin ________________________________

Actual Yield of recrystallized aspirin ________________________________

Percent Yield of recrystallized aspirin ________________________________

Melting Point of pure aspirin (literature) ________________________________

Melting Point of recrystallized aspirin ________________________________

Discussion Questions:

1. Determine the percentage yield of your crude product.

2. As in many organic reactions, the synthesis in this experiment is an equilibrium reaction. What steps could you take to improve the yield of aspirin in this particular experiment?

3. If the aspirin crystals were not completely dried before the melting point was determined, what effect would this have on the observed melting point?

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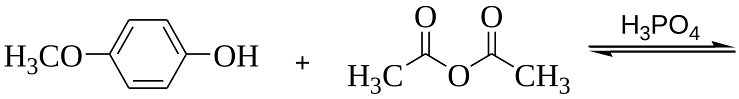
4. *(Optional)* Discuss the purity of your final product. How pure (or impure) is your aspirin based on literature values? Comment on how your IR spectrum parallels the spectrum of pure acetylsalicylic acid, making certain to LABEL and discuss all characteristic absorption peaks. You should turn in the labeled IR spectrum of your product with this report.

5. Consider the reaction shown below. Predict the product(s) of this reaction.



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Post-Lab Questions: Synthesis and Characterization of Aspirin

1. Determine the theoretical yield of aspirin that can be obtained from the addition of 2.0 grams of salicylic acid and 5.0 mL of acetic anhydride (density = 1.08 g/mL). What is the limiting reagent?

2. Determine the percentage yield of the reaction if 1.9 g of aspirin is obtained in this experiment.

3. What is the purpose of recrystallization?

4. Draw and name two different structural isomers for esters (RCOOR) with an empirical formula $C_3H_6O_2$.

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Experiment #16 – Introduction to IR and NMR Spectroscopy

Spectroscopy studies the light absorbing properties of matter. Since each compound has its unique molecular or ionic structure, its light absorbing properties will also be unique. Spectroscopy is often a quick method to obtain a lot of information about a compound's structure. Sometimes, with sufficient spectroscopic results, the compound's structure can be completely determined.

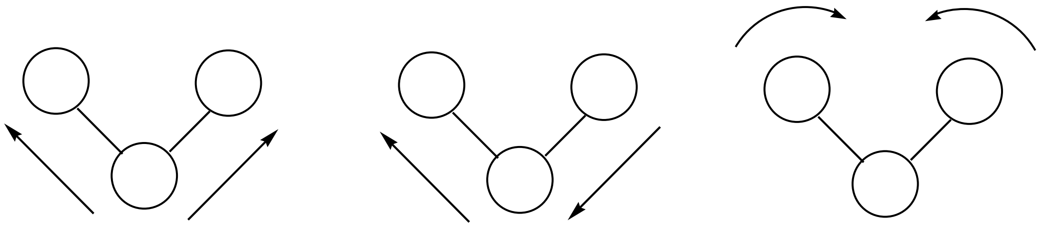
Depending on which region of the electromagnetic spectrum is being used for spectroscopy, the type of information obtained about the compound will vary. For example:

- Visible and ultraviolet spectroscopy: These wavelengths of light cause electrons to be promoted to higher energy orbitals. Thus, information about a molecule's orbitals and bonding can be obtained. You may remember an experiment investigating the visible light emission and absorption of hydrogen, helium, and mercury in your Chem 1A class.
- Microwave spectroscopy: Microwaves cause molecules to rotate. In fact, the microwave oven works by causing the fast rotation of water molecules. This rotational kinetic energy is observed during the heating of the water.
- Infrared spectroscopy (IR): Infrared light causes the bonds in molecules to vibrate.
- Nuclear magnetic resonance (NMR) spectroscopy: NMR spectroscopy uses radiowaves to obtain information about how atoms are connected in compounds.

The purpose of this experiment is to use IR and NMR spectroscopy to deduce the structure of a molecule. You will be given a list of possible compounds and structures and you will use the spectra obtained to decide which structure is the correct one while also explaining your reasoning.

The concepts of IR spectroscopy are more straightforward than NMR spectroscopy and they will be discussed first. Fundamentally, each type of bond in a molecule will absorb a characteristic frequency of IR light as it vibrates, and these frequencies can often be used to determine which types of bonds the molecule actually contains. These vibrations fall into several different categories as shown below for a generic three atom system:

Modes of Vibration



Symmetric stretching Antisymmetric stretching Scissoring

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The frequency of light that is absorbed is closely related to the strength of the bonds. An analogy is that of the stretching of a spring. If we imagine each bond as a spring, the frequency of vibration of a spring will be greater if the spring is stiffer. A stiffer spring is equated with a stronger bond; a stronger bond vibrates at a higher frequency.

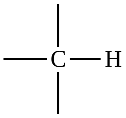
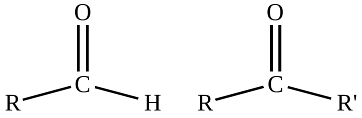
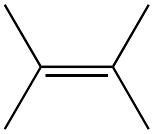
Planck’s equation relates the frequency, wavelength, and energy of light:

$$\Delta E = h\nu = hc/\lambda$$

where “h” is Planck’s constant (6.626×10^{-34} J s) , “ν” is the frequency of light, “λ” is the wavelength, and “c” is the speed of light (3.0×10^{8} m/s). Notice that:

$$\nu = c/\lambda$$

In IR spectroscopy, wavelengths were measured in microns (μm) and frequency was measured in cm^{-1} (called “wavenumbers”). Your measurements will be made in wavenumbers (note that wavenumbers, $1/\lambda$ or cm^{-1}, and frequency, ν, are directly proportional). Some typical frequencies in wavenumbers for the stretching of common types of bonds are given below:

Alcohols	RO-H	3200-3650 cm^{-1}
Amines	R_2N-H	3300-3500 cm^{-1}
Alkanes		2840-3000 cm^{-1}
Carbonyl group in aldehydes and ketones, C=O,		1690-1750 cm^{-1}
Alkenes		1620-1680 cm^{-1}

In general, the symmetric bond stretching frequencies are more clearly shown in the IR spectrum and found in the 4000-1500 cm^{-1} region. The other types of vibrations, antisymmetric stretches and scissorings, are generally seen in a complex region below 1500 cm^{-1} called the “fingerprint region.” However, since the IR spectrum of each unique molecule should also be unique, the entire spectrum actually serves as a “fingerprint” of the molecule. The spectrum can be used to identify the types of bonds in the molecule, like the examples given above, or, if the spectrum of your compound matches that of a known compound, you can conclude that your compound and the known are the same.

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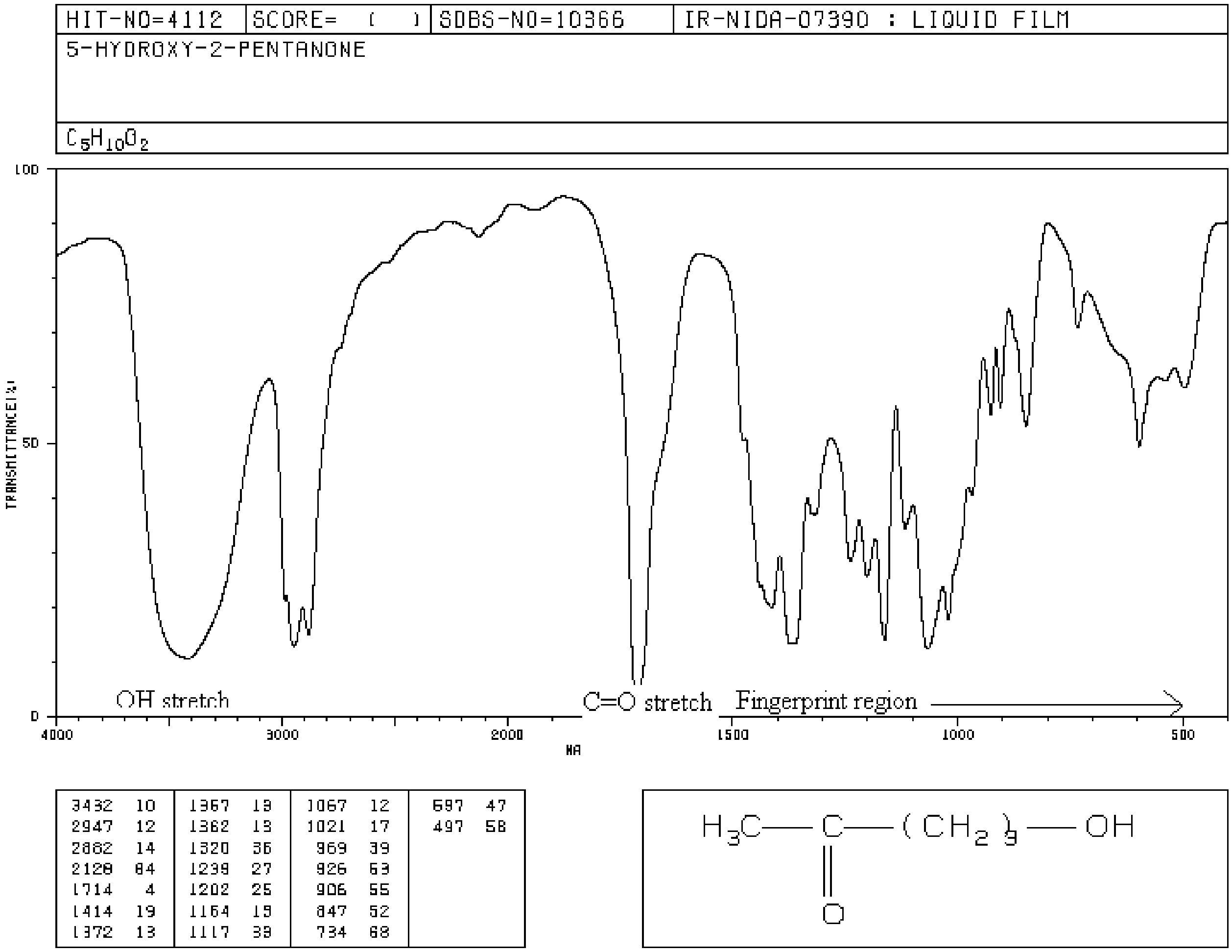
A website is available which gives thousands of IR and NMR spectra for common compounds. The spectral examples given in this experiment come from that website and its address is:

https://sdbs.db.aist.go.jp/sdbs/cgi-bin/cre_index.cgi

Given below is the IR spectrum of the indicated molecule:



HIT-NO=4112	SCORE= ( )	SDBS-NO=10366	IR-NIDA-07390 : LIQUID FILM
5-HYDROXY-2-PENTANONE			
$C_5H_{10}O_2$			

3432	10	1367	13	1067	12	697	47
2947	12	1362	13	1021	17	497	58
2882	14	1320	36	969	39		
2128	84	1239	27	926	63		
1714	4	1202	25	906	55		
1414	19	1164	19	847	52		
1372	13	1117	33	734	68		

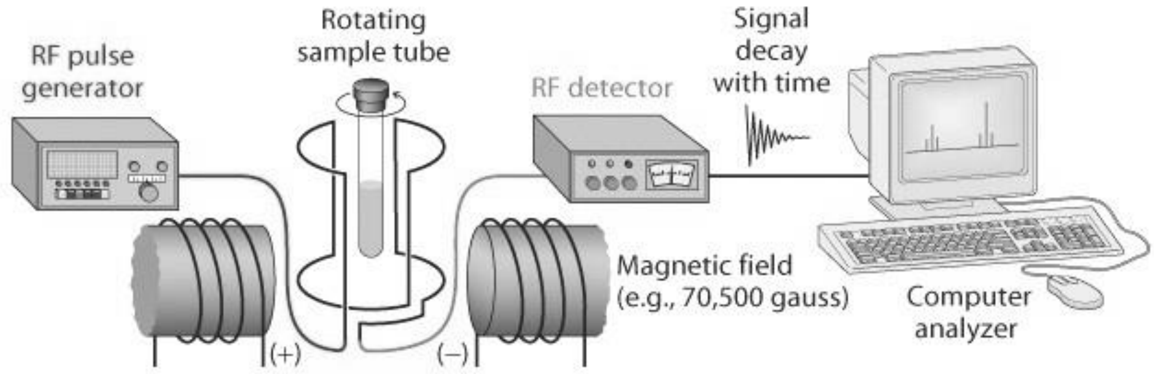
$H_3C-C(=O)-(CH_2)_3-OH$

The two most outstanding features of this spectrum are the very broad and strong absorption centered at 3432 cm^{-1} for the stretch of the O–H bond (the broadness is caused by hydrogen bonding, especially with traces of water) and the carbonyl stretch (C=O), strong and narrow, at 1714 cm^{-1}. The fingerprint region is shown below 1500 cm^{-1}.

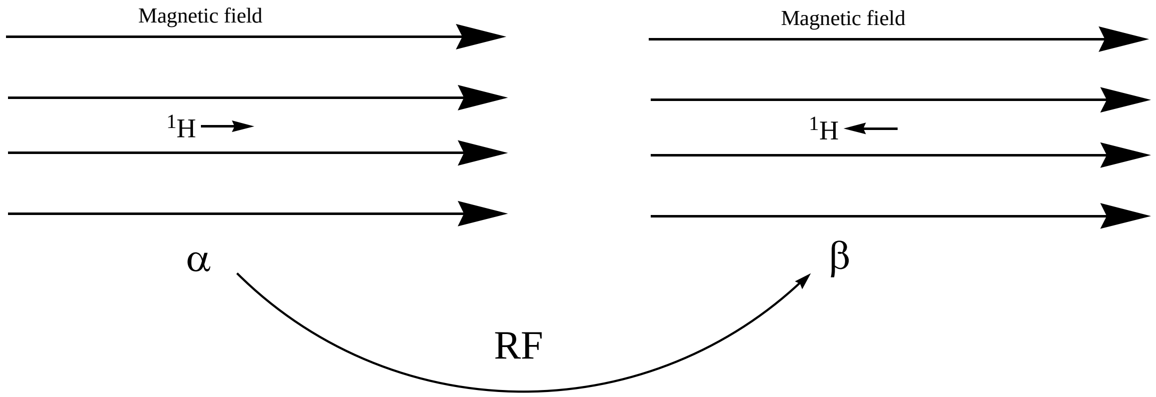
The compounds that you will be given as unknowns will contain either alcohol bonds, O–H, or carbonyl bonds, C=O, so you should see an absorption similar to one in the spectrum above.

The underlying principles of NMR spectroscopy are much less clear-cut than those of IR spectroscopy. NMR spectroscopy “observes” nuclei that have an overall net spin. The concept of the spin in a nucleus is fundamentally the same as the spin of electrons. Electrons have spins of +½ or –½ (↑ or ↓); protons and neutrons in the nucleus have analogous spins. In each case, spins can be paired or unpaired. Although it is fairly easy to predict the pairing of electrons in an atom, this is not easily done for the pairing of spins in a nucleus. However, any nucleus that has an overall net spin can be observed in an NMR experiment. The most useful nucleus for observation in an NMR experiment is the nucleus of the major isotope of hydrogen, ^{1}H, which consists of one proton and, therefore, has a spin of ½. It is interesting to note that deuterium, ^{2}H, has a spin of 1, i.e., both the proton and neutron in the nucleus contribute spin. Several other important nuclei in NMR spectroscopy are ^{13}C, ^{19}F, ^{31}P (spins of ½) and ^{11}B (spin of 3/2) and ^{14}N (spin of 1).

The following discussion of the principles of NMR will be given for ^{1}H, but a similar explanation can be developed for any nucleus with a spin. A schematic of an NMR spectrometer is shown below (taken from Volhardt and Schore, Organic Chemistry, 4th Edition). Pay particular attention to the fact that there is an extremely powerful magnet and a radiofrequency (RF) pulse generator.



When a compound containing hydrogen is placed in a magnetic field, the spin of the ^{1}H nuclei can be aligned parallel to the lines of the magnetic field (lower energy, called α) or anti-parallel (higher energy, called β):



The applied radiofrequency energy from the generator will cause the spins to be promoted to the higher energy state.

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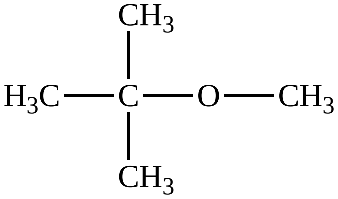
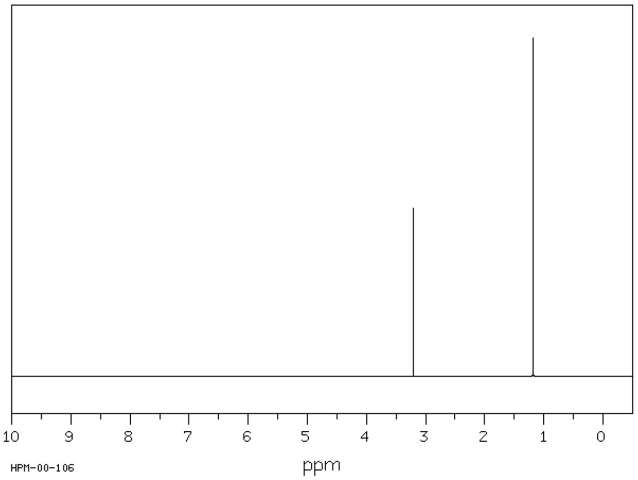
Now, consider the fact that a given molecule will generally have several different types of hydrogens in it. For example, consider the molecule methyl t-butyl ether:



$$H_3C{-}C(CH_3)_2{-}O{-}CH_3$$

There are two types of hydrogen: the three hydrogens bonded to the methyl group (CH_3) attached to the oxygen and the nine H’s bonded to the CH_3 groups attached to the central carbon. These will undergo the $\alpha \rightarrow \beta$ transition at two different frequencies. In fact, imagine that the molecule is subjected to a pulse of all possible radio frequencies. This causes all ^1H nuclei to be in the β-state and, once the pulse is ended, they will decay to the α state and emit their characteristic radio frequencies. These emissions will be picked up by the RF detector. These signals are then manipulated mathematically and plotted, yielding the following NMR spectrum for our compound:



Notice that the scale is labeled “ppm,” commonly called a δ (delta) value or “chemical shift.” The zero value on the scale represents the frequency at which a standard compound, $(CH_3)_4Si$ (tetramethylsilane, or TMS), would absorb. This is used as a reference point.

The value of the **chemical shift (δ)** is related to the electron density around the hydrogen atom. δ-values are larger if the electron density is lower.

Electronegative atoms or electron withdrawing groups near the carbon to which the hydrogens are bonded will cause a frequency with a higher δ-value. In the spectrum above, the peak at 3.4 will correspond to the methyl hydrogens for the group bonded to O.

Some typical chemical shift ranges are shown:

RCH_3 δ=0.8-1.0
RCH_2R' δ=1.2-1.4
R_3CH δ=1.4-1.7
RCH_2Cl δ=3.6-3.8
RCH_2OH δ=3.3-4.0 (for CH_2 hydrogens, OH hydrogen is often not seen)
ROH, if seen, δ=0.5 to 5 (the large range caused by hydrogen bonding effects)
Ketone, $RCOCH_3$, δ=2.1-2.6
Aldehyde, RCOH, δ=9.5-10.0
Aromatic, i.e., H attached to phenyl ring, δ=6.0-9.5

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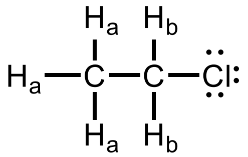
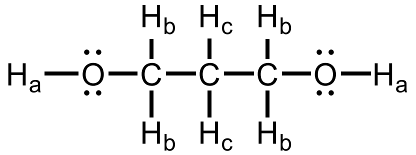
The location in terms of δ-values can often give a good idea of what is directly bonded to the carbon on which the observed hydrogen(s) are located.

Although the chemical shift values can give an idea of types of bonds that are present in the molecule—similar to the information obtained in the IR spectrum—even more important is the effect that hydrogens on neighboring atoms will have on the frequencies observed. To see this effect, however, these neighboring hydrogens **must be chemically different.** So, before discussing these effects, you must be able to recognize when hydrogens are the same and when they are different. Four examples are given below, with each unique type of hydrogen labeled with a different letter:

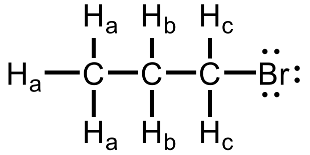
a b
CH_3CH_2Cl



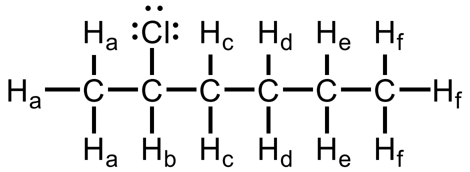
a b c b a
$HOCH_2CH_2CH_2OH$



a b c
$CH_3CH_2CH_2Br$



a b c d e f
$CH_3CHClCH_2CH_2CH_2CH_3$



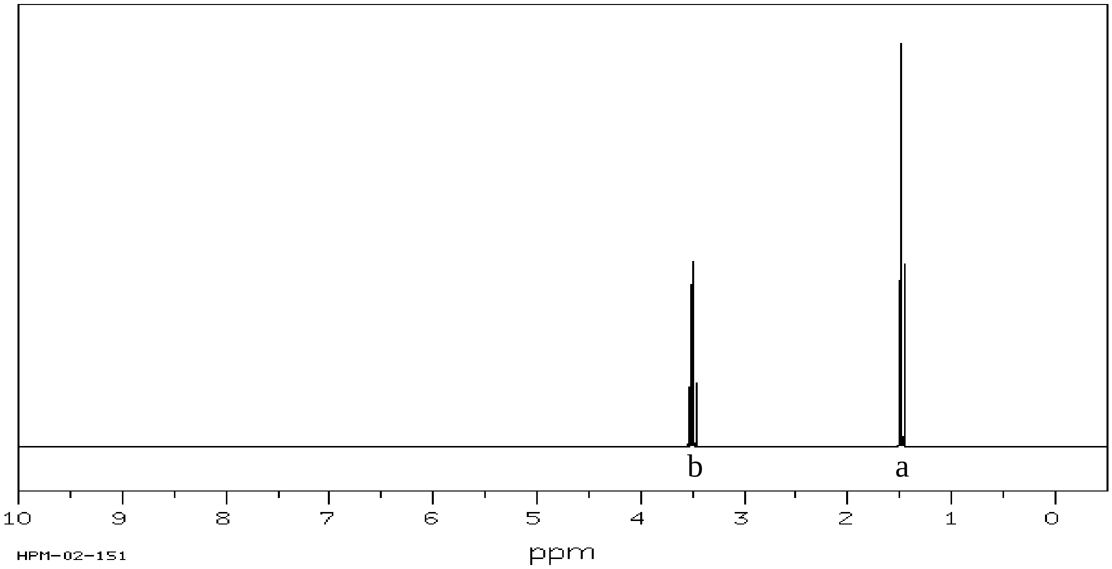
For hydrogens to be the same, they must be in completely equivalent environments. Therefore, in the second example above, the CH_2's are not all the same; the one in the middle is not in the same environment as those labeled at "b" since "b" is immediately adjacent to an OH and "c" is not.

The equivalence or non-equivalence of hydrogens is critical in NMR spectroscopy since the hydrogens on neighboring carbon atoms will split the frequency signal into multiple peaks. If there is one neighboring hydrogen the signal will be split into two peaks, called a doublet; two neighboring hydrogens will split the signal into three peaks, called a triplet; three into a quartet; four into a pentet, etc. These are called, generically, "multiplets." However, the intensities of these split peaks are not equivalent, except for the doublet. A triplet is 1:2:1, a quartet is 1:3:3:1, a pentet is 1:4:6:4:1. If you are familiar with Pascal's triangle, you will see that these intensities follow the triangle's pattern. Some of the corresponding spectra for the compounds follow.

Name: ______________________________ Section: ____________________

a b

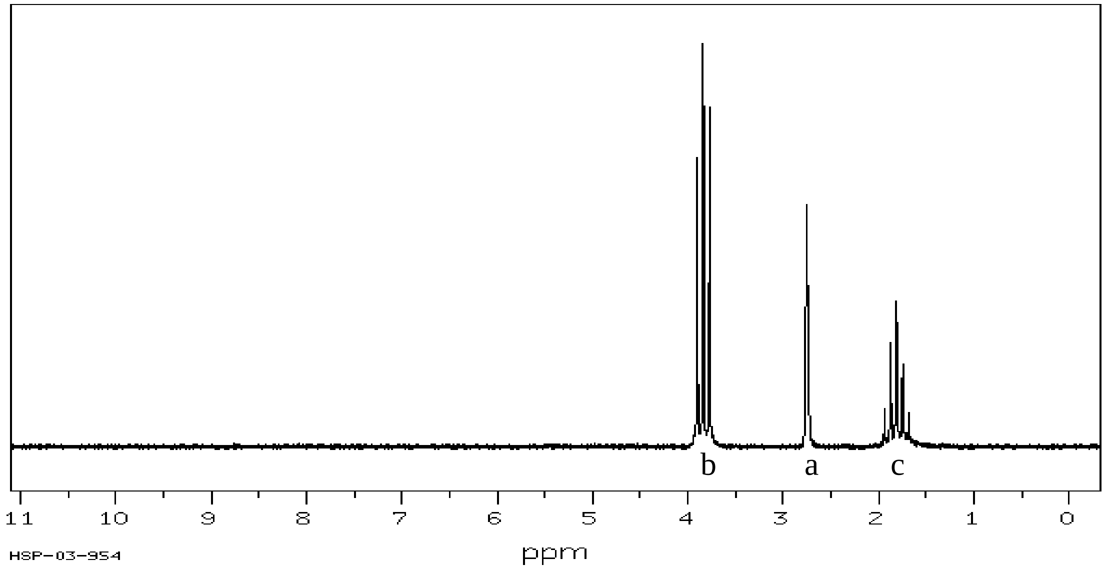
CH_3CH_2Cl (magnet strength is 70,500 gauss)



Carefully examine the spectrum above to see the relation of the splitting patterns to the structure of chloroethane. The quartet labeled “b” is caused by the three neighboring hydrogens “a” and the triplet “a” caused by the two neighboring hydrogens labeled “b.”

a b c b a

$HOCH_2CH_2CH_2OH$ (magnet strength is 21,000 gauss)



Note that in the spectrum above, the triplet (δ=3.8) is wider than the triplet from the previous spectrum, making it easier to identify the three peaks. This is an effect caused by the lower magnetic field strength. The pentet labeled “c” is caused by the four neighboring hydrogens “b.” Note that the hydrogens labeled “a” form a singlet. As a result of hydrogen bonding, alcohol hydrogens always appear as singlets and do not cause other hydrogens to split.

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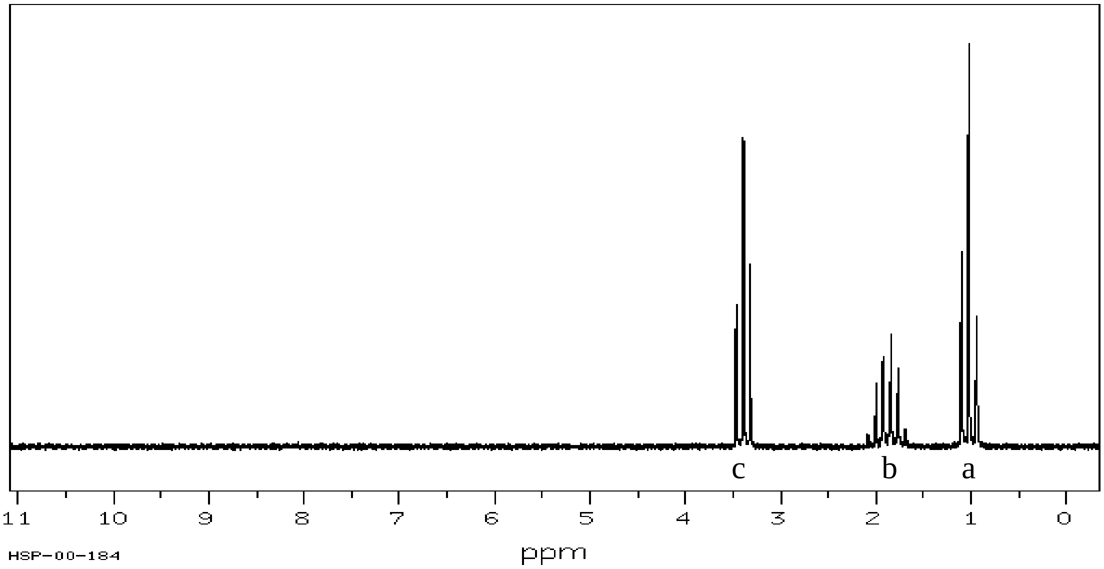
Analysis becomes more complex when hydrogens are split by neighboring hydrogens, but the neighbors are non-equivalent. Consider the case shown below:

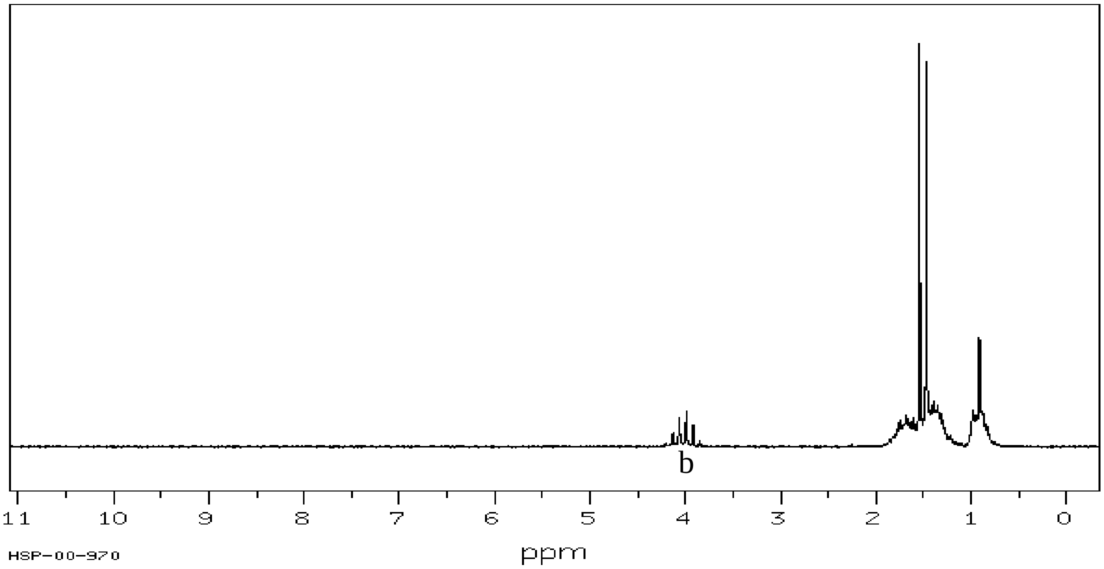
a b c
$CH_3CH_2CH_2Br$



The hydrogens labeled “b” are split on one side by “a” and then further split by “c,” the overall result being a quartet of triplets or a triplet of quartets, more easily called simply a “multiplet.”

Finally, extremely complex is:

a b c d e f
$CH_3CHClCH_2CH_2CH_2CH_3$



Its interpretation is beyond this experiment, but the hydrogen labeled “b” is found at a value of δ=4 since there is an electronegative chlorine also attached to the carbon.

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Finally, there are two more issues to discuss concerning the NMR spectra: the area under the signals, called the **integral**, and what **solvents** are used to dissolve samples for analysis.

Each signal in an NMR spectrum represents a certain number of hydrogen atoms. If the area under the entire signal (all of the peaks in a "multiplet") is measured, this is called the integral and this area is proportional to the number of hydrogens represented by the signal. For example, in the first spectrum above, CH_3CH_2Cl, the relative integrals would be 2:3 for b:a. The NMR spectrometer will automatically measure the integrals of the signals and list them, but these will not normally be whole numbers. The ratios should be close to the whole number ratios, e.g., you might see 0.386:0.570, close to 2:3, but not exactly equal.

And last of all is the consideration of the solvent. Since NMR solutions are not very concentrated and consist mainly of solvent, any hydrogens in the solvent will give extremely strong NMR signals which may obscure signals from your compound. Fortunately, almost all commonly used solvents can be purchased with deuterium (D) replacing hydrogen and since D is twice the mass of H, the deuterium signal will not appear in your spectrum. For example, deuterated chloroform, $CDCl_3$, is commonly used, a solvent which dissolves most organic compounds and is relatively inexpensive.

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EXPERIMENTAL PROCEDURE

Paper Unknowns:

1. Analyze the Paper Unknowns on the next page. Fill in the missing carbon and hydrogen atoms. Identify whether the compound has an alcohol (R–OH) or carbonyl (R–C=O). Label and count the hydrogen atom types starting with "a", "b", etc., be aware some hydrogens may have identical neighbors. Predict the expected splitting patterns; singlet, doublet, triplet, quartet, multiplet. Take note of nearby electronegative atoms and expected chemical shifts.

2. Obtain a printout of the IR Spectrum and NMR spectrum of your unknown from your instructor. Analyze the spectrums. Use the list of possible paper unknown compounds and their structures and an analysis of the spectra to decide which compound on the list is your unknown. You will submit a brief analysis which should consist of your choice of the correct compound and a complete explanation of your reasoning involved in coming to that conclusion. This analysis should include interpretation of the splitting patterns in the NMR spectrum, the correspondence of the δ-values to the types of hydrogen in the compound, and an interpretation of the IR spectrum, identifying the corresponding absorptions for the functional groups.

Liquid Unknowns:

1. Draw a 3D structural picture for each of the Liquid Unknowns. Identify whether the compound has an alcohol (R–OH) or carbonyl (R–C=O). Label and count the hydrogen atom types starting with "a", "b", etc., be aware some hydrogens may have identical neighbors. Predict the expected splitting patterns; singlet, doublet, triplet, quartet, multiplet. Take note of nearby electronegative atoms and expected chemical shifts.

2. Learn how to use the IR instrument in our Organic Chemistry lab room PS 103.

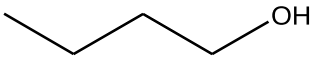
3. Run an experimental IR Spectrum for a drop of your liquid unknown. Clean up after use.

4. Obtain a printed NMR spectrum of that same unknown from your instructor.

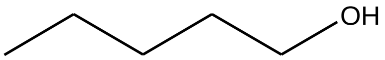
5. Analyze the IR and NMR Spectrums. Use the list of possible liquid unknown compounds and their structures and an analysis of the spectra to decide which compound on the list is your unknown. You will submit a brief analysis which should consist of your choice of the correct compound and a complete explanation of your reasoning involved in coming to that conclusion. This analysis should include interpretation of the splitting patterns in the NMR spectrum, the correspondence of the δ-values to the types of hydrogen in the compound, and an interpretation of the IR spectrum, identifying the corresponding absorptions for the functional groups.

Name: ________________________________ Section: ____________________

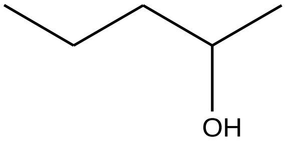
Part A: Paper Unknowns



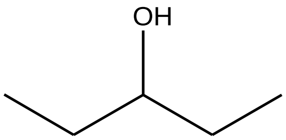
1-butanol



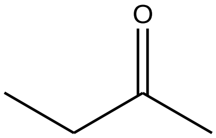
1-pentanol



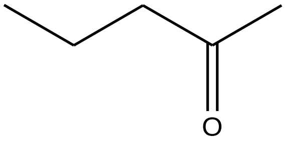
2-pentanol



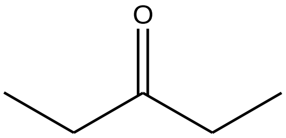
3-pentanol



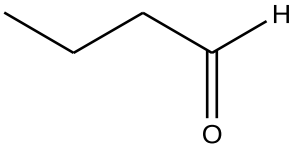
2-butanone



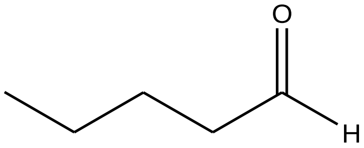
2-pentanone



3-pentanone



butanal



pentanal

Name: ______________________________ Section: ____________________

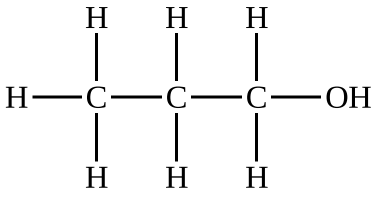
Part B Liquid Unknowns

Name of Solvent	Formula	Draw the 3D structure
acetone or 2-propanone	C_3H_6O	
ethyl acetate or ethyl ethanoate	$C_4H_8O_2$	
methanol	CH_4O	
ethanol	C_2H_6O	
1-propanol	C_3H_8O	
2-propanol or isopropanol	C_3H_8O	
methyl acetate or methyl ethanoate	$C_3H_6O_2$	
methyl ethyl ketone or 2-butanone	C_4H_8O	

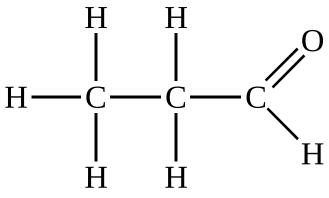
Name: ______________________________ Section: ____________________

Post-Lab Questions: Introduction to IR and NMR

Consider the following two molecules:



1-propanol

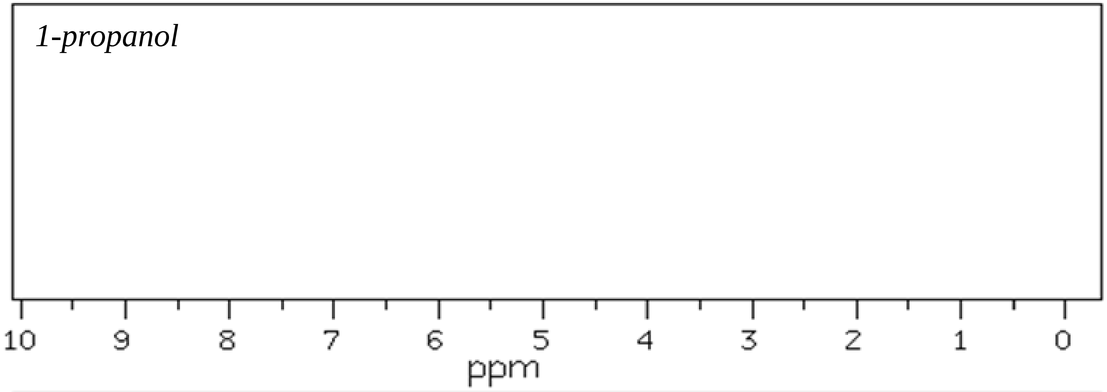


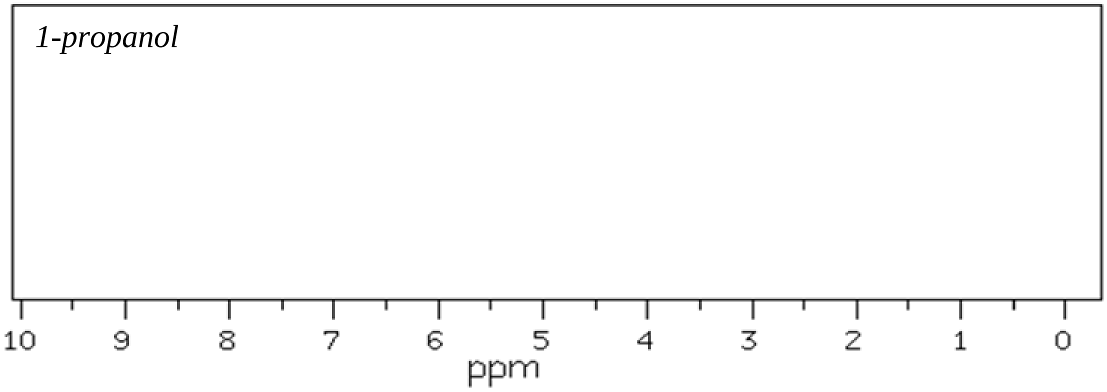
propanal

1. What should you see in their infrared spectra that should allow them to be distinguished?

2. Label each of the hydrogen atoms in the molecules above with the letters “a,” “b,” “c,” etc. to indicate all of the different types of hydrogen.

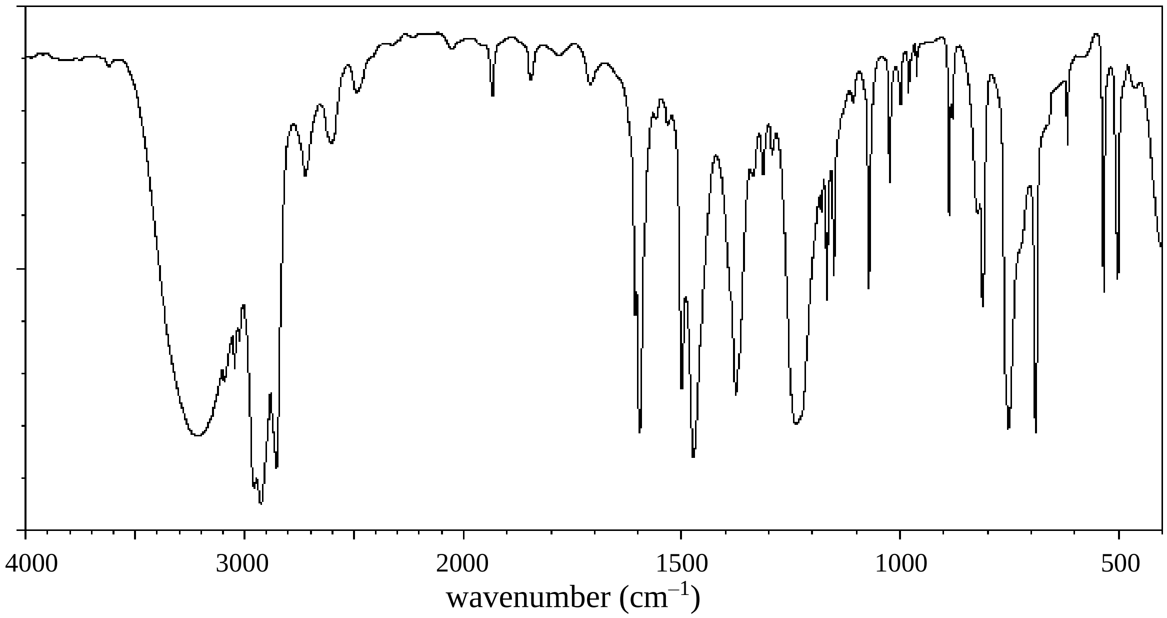
3. For each of the molecules, draw what you would expect the ^{1}H NMR spectrum to look like. Label each signal with the letters that you used in question 2 (“a,” “b,” “c,” etc.). Be sure to place the signals in the appropriate chemical shift order and to draw ***and label*** the appropriate splitting pattern:

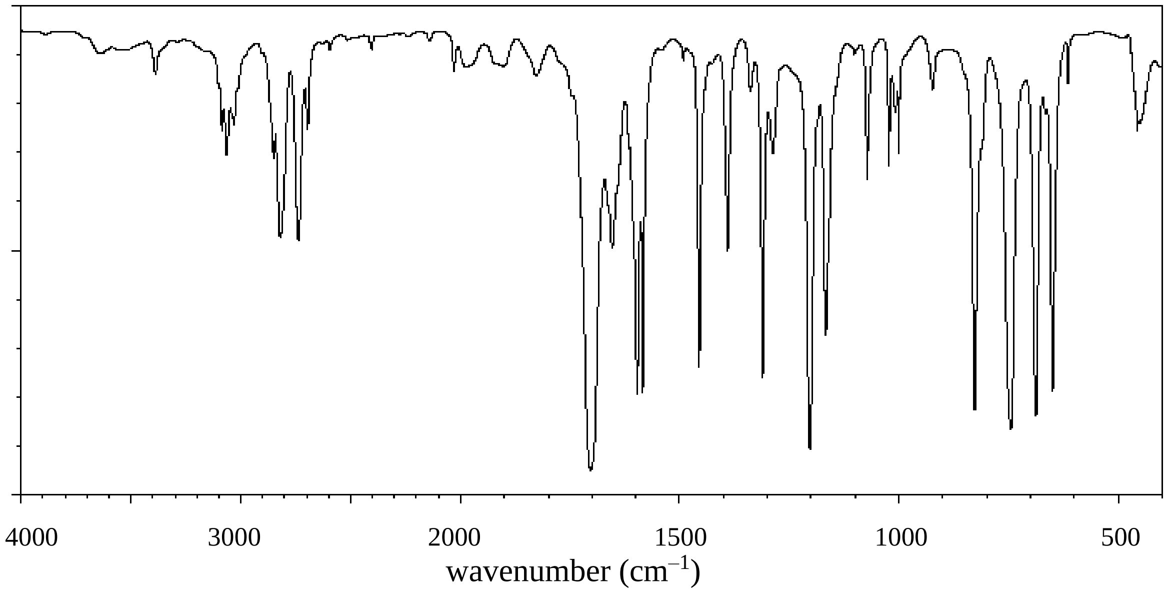




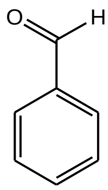
Name: ______________________________ Section: ____________________

4. Consider the two IR spectra shown below. One spectrum is characteristic of benzaldehyde; another spectrum is characteristic of phenol. Which spectrum belongs to which compound? Briefly explain, making sure to identify relevant absorption peaks.



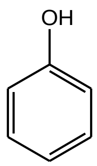


Compound I



benzaldehyde

Compound II



phenol

Name: ______________________________ Section: ____________________

Error Analysis, Statistics and Graphing Workshop

Percent error:

The error of a measurement is defined as the difference between the experimental and the true value. This is often expressed as **percent (%) error**, which is calculated as:

$$\text{Percent error} = \left|\frac{\text{Experimental} - \text{True}}{\text{True}}\right| \times 100\ \% \qquad (1)$$

Note: At times a true value may not be known or possible. If two experimental values are determined and the true value is unknown, then the percent difference may be calculated. The percent difference is the absolute value of the difference between the two experimental values divided by the average value and multiplied by 100.

$$\%\ \text{difference} = \frac{|\text{Value 1} - \text{Value 2}|}{(\text{Value 1} + \text{Value 2})\ /\ 2} \times 100\ \%$$

In chemical measurements, we try to eliminate errors, which can be divided into two broad types: systematic and random. *Systematic error* occurs regularly and predictably because of faulty methods or sampling techniques, defective instrumentation or calibration, and/or incorrect assumptions. *Random error* is more difficult to define and is governed by chance. Examples include a weighing error due to air currents or changes in temperature near a balance and line current fluctuations for electronic instrumentation. Systematic errors always affect the measured quantity in the same direction, while random errors can make the measured quantity either too large or too small.

Accuracy is the closeness of agreement between a measured value and the true (or accepted) value. True values can never be obtained by measurement. However, we accept values obtained by skilled workers using the best instrumentation as true values for purposes of calculation or for judging our own results.

Precision describes the reproducibility of our results. A series of measurements with values that are very close to one another is a sign of good precision. It is important to understand, though, that good precision does NOT guarantee accuracy!

Name: ______________________________ Section: ____________________

Standard Deviation:

The ***standard deviation*** of a series of measurements including at least 6 independent trials may be defined as follows: let x_m represent a measured value, n be the number of measurements, and $\bar{x}$ be the average or mean of the various independent trials or measurements. Then d is the average deviation:

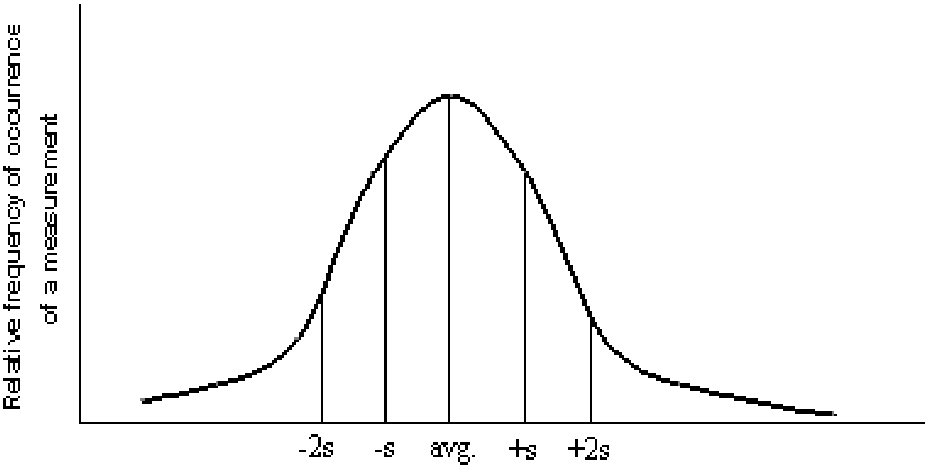
$$d = x_m - \bar{x} \tag{2}$$

and the standard deviation, *s*, is defined by:

$$s = \sqrt{\frac{\sum d^2}{n-1}} \tag{3}$$

where $\sum d^2$ refers to the sum of all the values of d^2.

The standard deviation is used to indicate precision when a large number of measurements of the same quantity are subject to random errors only. We can understand the meaning of s if we plot the number of times a given value is obtained (on the y-axis) versus the values x_m (on the x-axis). Such a normal distribution curve is bell-shaped with the most frequent value being the average value $\bar{x}$.



Most measurements result in values near $\bar{x}$; 68% of the measurements fall within one standard deviation while 95% of the measured values are found within 2s of $\bar{x}$. The value of 2s is called the uncertainty of the measurement. If we report our value of the measurement as the range $\bar{x} \pm 2s$, we are saying that $\bar{x}$ is the most probable value, and 95% of the measured values fall within this range.

$$\text{range} = \bar{x} \pm 2s \tag{4}$$

Name: ________________________________ Section: ____________________

Q Test:

For most of the experiments in this course, the standard deviation is impossible to calculate because we perform too few measurements of a particular quantity. When there are such few measured values (< 6), the ***Q Test*** is used to decide whether to reject suspected “bad” values as outliers.

$$Q = \left|\frac{\text{suspect} - \text{nearest}}{\text{largest} - \text{smallest}}\right| \tag{5}$$

n **(# of measurements)**	**3**	**4**	**5**	**6 or more**
Q Test **value (90% probability)**	0.94	0.76	0.64	Do not use

If Q for a set of data is larger than those listed in the table, the suspect value may be rejected, and the average of the other values is reported. Please note that the sign of Q is NOT important since we are using the absolute value.

Graphical Representation of Data and the Use of Excel®:

Scientists answer posed questions by performing experiments which provide information about a given problem. After collecting sufficient data, scientists attempt to correlate their findings and derive fundamental relationships that may exist between the acquired data. Whether a set of measurements or variables are correlated can be examined by constructing a graph and calculating the coefficient of determination (also known as r^2). Microsoft Excel® is a program commonly used to construct a graph and calculate r^2. Instruction on how to use Excel® for graphing is given later.

Graphing:

Graphical representations of data illustrate relationships among data visually. A graph is a diagram that represents the variation of one factor in relation to one or more other factors. These variables can be represented on a coordinate axes.

The vertical axis is the y-axis (or ordinate), and the horizontal axis is the x-axis (or abscissa). When plotting a certain variable on a particular axis, experiments are normally designed so that you vary one property (represented by the *independent variable*) and then measure the corresponding effect on the other property (represented by the *dependent variable*).

All graphs should conform to the following guidelines:

1. They should have a descriptive title.
2. The independent variable is conventionally placed on the horizontal axis; the dependent variable is plotted on the vertical axis.
3. Label both the vertical and horizontal axes with units clearly marked.
4. The scale chosen for the data should reflect the precision of the measurements. For example, if temperature is known to be +0.1 °C, you should be able to plot the value this closely. Moreover, the data points should be distributed so that the points extend throughout the entire page (as opposed to a small portion of the paper).
5. There should be a visible point on the graph for each experimental value.

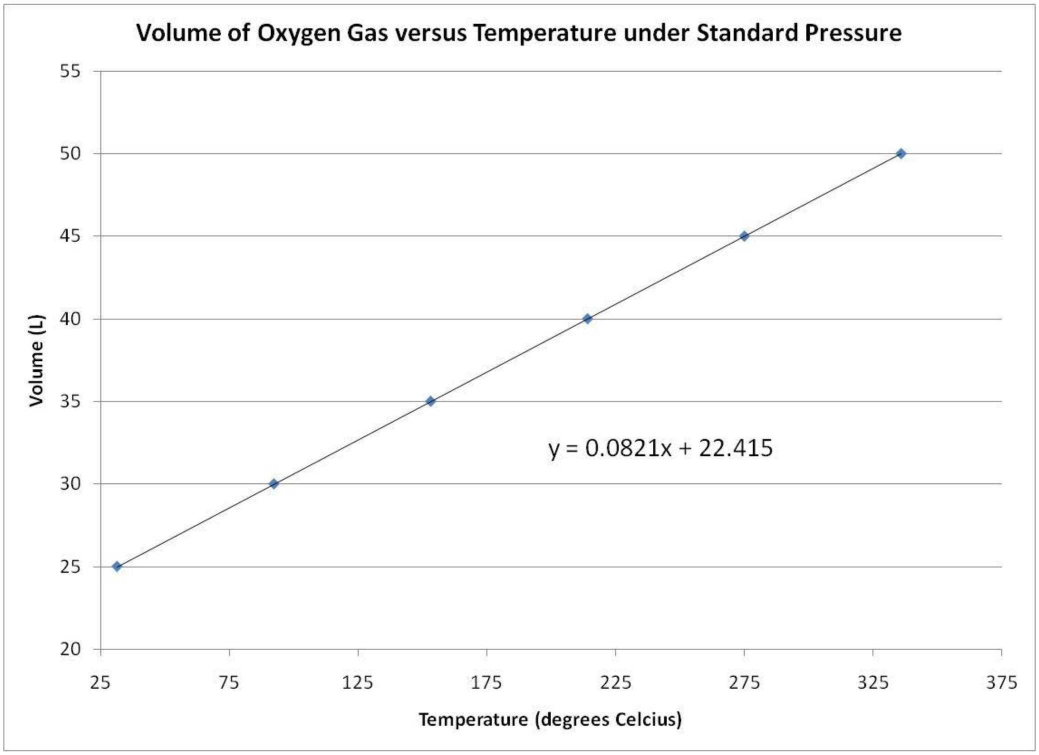
Name: ______________________________ Section: ____________________

Linear Graph:

Let us first examine a direct function involving a linear graph. Consider the following measurements made of an oxygen sample under standard pressure:

Temperature (ºC)	**Volume (L)**
31.49	25.00
92.38	30.00
153.28	35.00
214.18	40.00
275.08	45.00
335.97	50.00

Using graph paper or any graphing program such as Microsoft Office Excel®, one can first construct a plot of the data, where volume is determined to lie on the y-axis, and temperature is plotted on the x-axis. Once the data is plotted, a best-fitting line is constructed, and an equation of the line in slope-intercept form ***y = mx + b*** is formulated, where *m* = slope and *b* = y-intercept. That is,



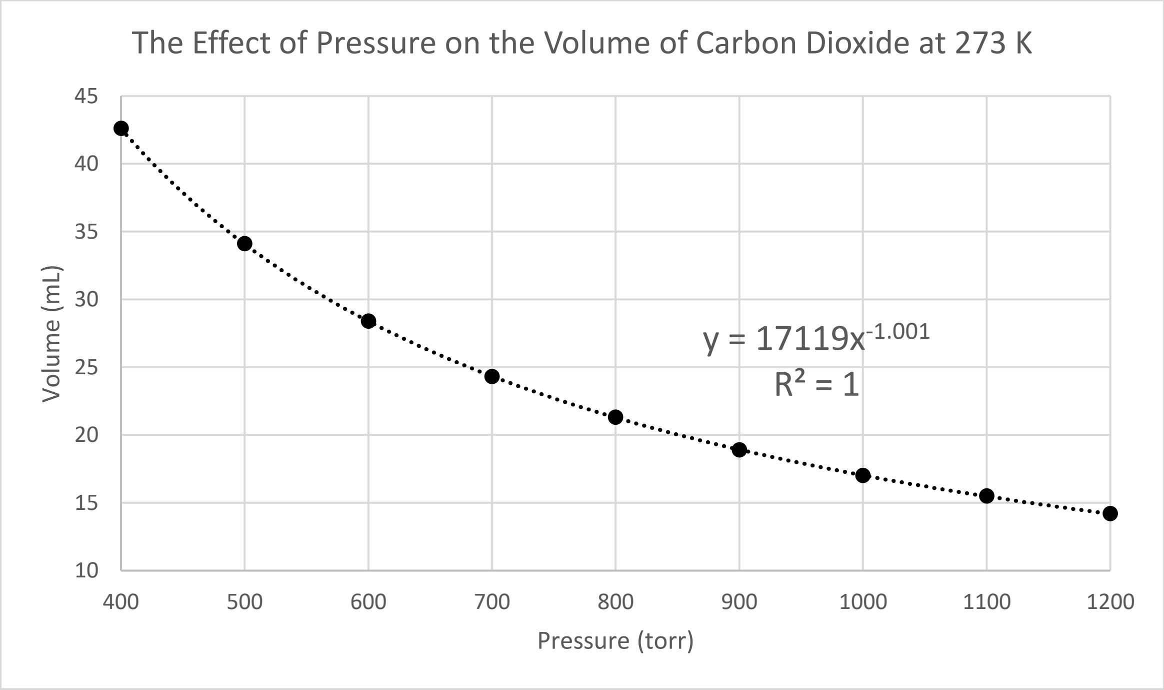
Name: ______________________________ Section: ____________________

Non-Linear Graph:

Now examine an indirect function involving a hyperbola. Consider the following measurements made of a carbon dioxide gas sample at 273 K:

Pressure (torr)	Volume (mL)
400	42.6
500	34.1
600	28.4
700	24.3
800	21.3
900	18.9
1000	17.0
1100	15.5
1200	14.2

Once again, using graph paper or any graphing program such as Microsoft Office Excel®, one can construct a plot of the data, where volume is determined to lie on the y-axis, and pressure is plotted on the x-axis.



As depicted in the graph above, some chemical relationships are not linear; that is, there are no simple linear equations to represent such relationships. Instead, a plot of data for this kind of relationship gives a curved (non-linear) fit. Such a graph is useful in showing an overall chemical relationship, although the slope and the y-intercept are NOT relevant to its interpretation.

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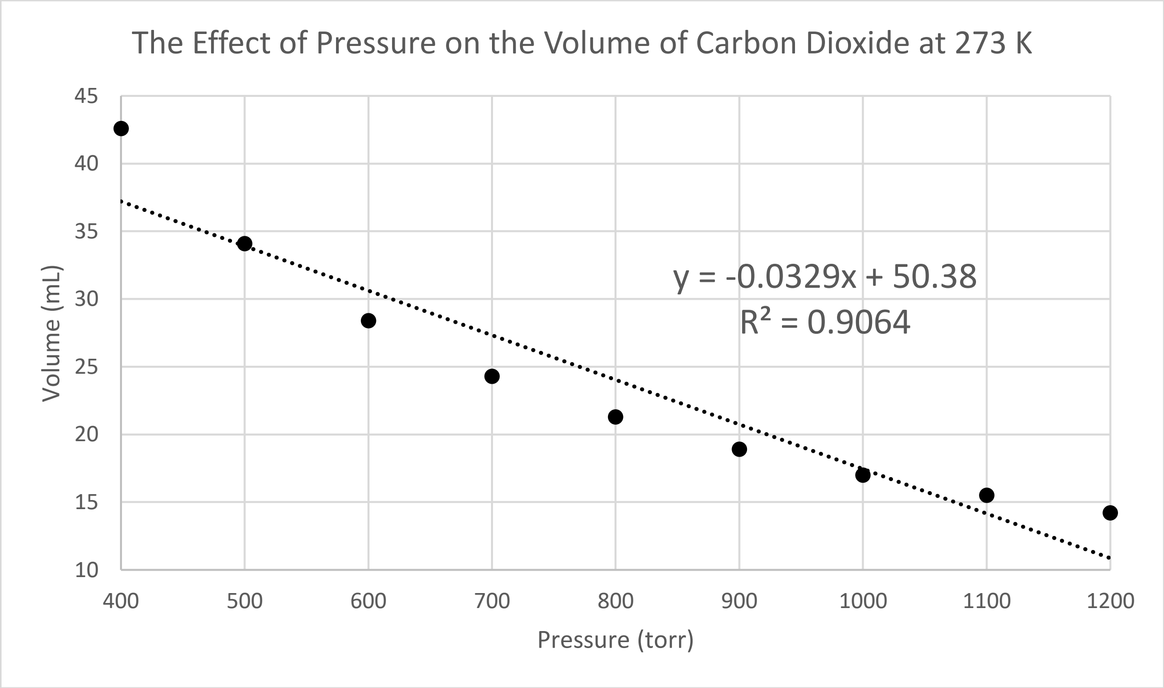
Coefficient of Determination, r^2: Is x correlated with y?

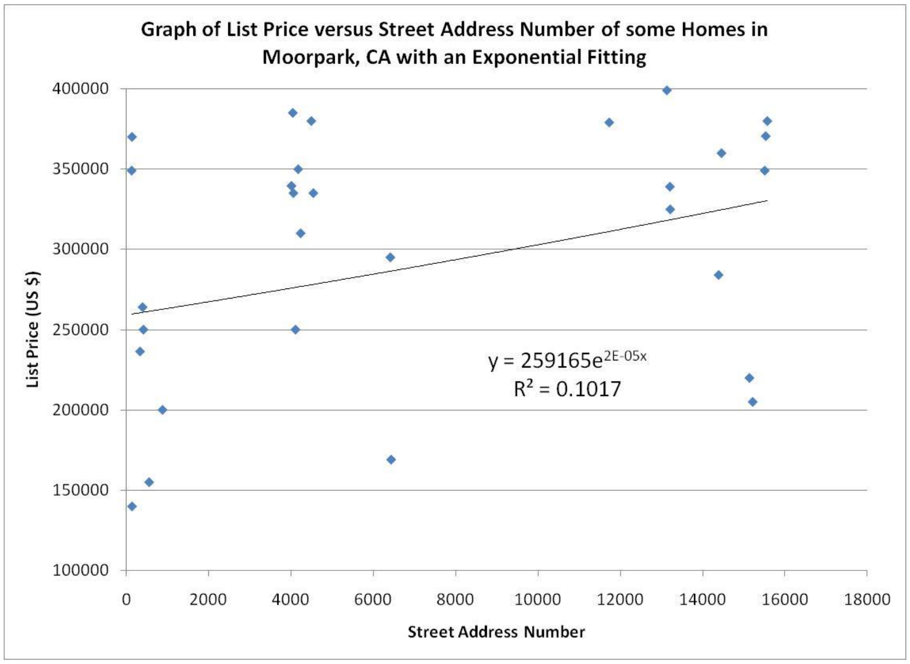
A set of (x,y) values are not always correlated in a linear or any other models/fittings. The coefficient of determination or the r^2 (or the Excel® function **RSQ)** is a measure of the correlation or linear dependence (in the case of a linear fitting) between the (x,y) variables. This coefficient of determination indicates how strongly a set of x values correlate with the corresponding set of y values. The r^2 value ranges from 0 to 1. A value of 1 means that data set perfectly fits a linear model or equation and value of 0 means that there is no correlation between x and y. A value of 0.8 means that 80% of the data fit the model/fitting.

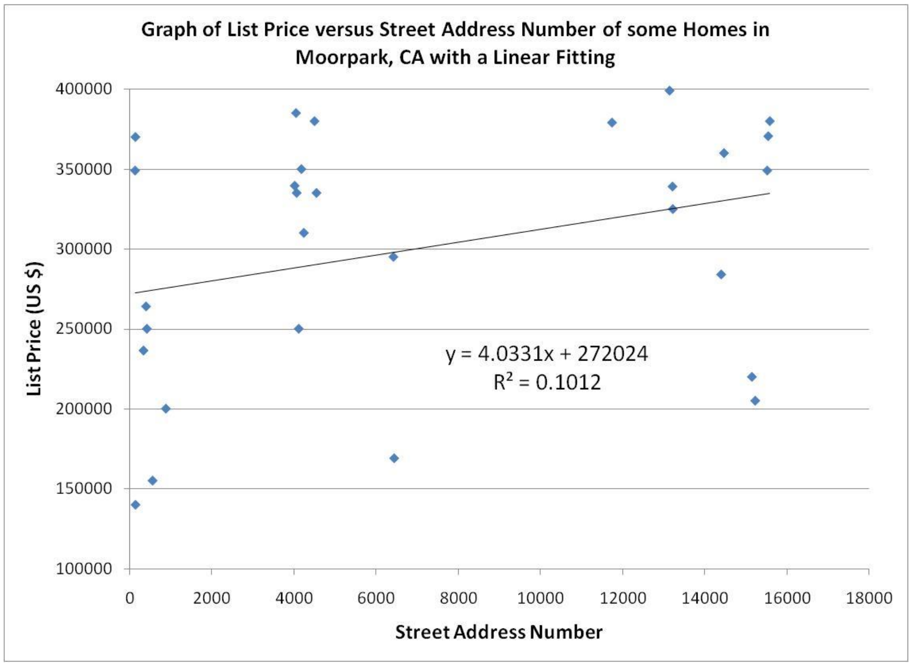
Let's examine the two graphs above (Temperature vs Volume and Pressure vs Volume). The r^2 value for the linear fitting of Temperature vs Volume is 1 (a perfect fit!). If a linear trendline is used on the Pressure vs Volume graph, an r^2 value of 0.9064 (below) is obtained. Pressure and volume, in this case, are correlated but a linear model might not be the best fit. If a power trendline is used (see previous page), an r^2 value of 1 is obtained. This means that x and y are correlated and a power trendline better explains the correlation than a linear trendline.



One can also have a data set that is not correlated to each other. Note the two graphs below. The data gives the list prices of some of the homes for sale in Moorpark, CA and their corresponding street address number. Since street address numbers are not unique to a neighborhood, we can easily conclude that there should not be any correlation between the two variables. The r^2 for the exponential and linear fittings are 0.1017 and 0.1012, respectively. These values are significantly lower than the ones discussed above. These low

r^2 values demonstrate that there is no correlation (linear nor exponential) between list price and street address number:





Excel® calculates the r^2 value by taking the square of r (also known as Pearson Product Moment Correlation Coefficient), defined by equation 5 below. An r^2 value equal to or greater than 0.99 "generally" means that the data has a "good" fitting to the trendline equation.

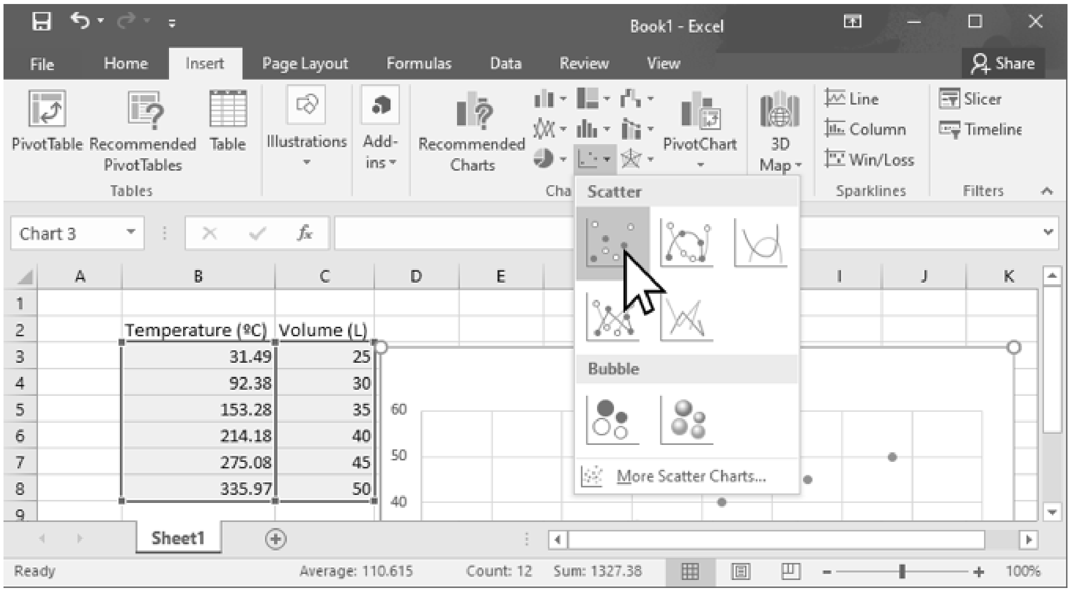
$$r = \frac{\sum (x-\bar{x})(y-\bar{y})}{\sqrt{\sum (x-\bar{x})^2 (y-\bar{y})^2}} \qquad (6)$$

Name: ________________________________ Section: ____________________

Excel® Procedure

Note that various versions of Excel® may function a bit differently from the directions outlined below (which work on department-owned laptop computers):

Put the title for your x-axis (include units) in one Excel® cell (box). In the cell to the right, put the title for your y-axis. Using these boxes as headings, input the numeric data (like a table) in the cells under these titles (each box should contain one number; each row represents one data point in x,y format). Click and drag your mouse to highlight just the numeric boxes. From the "Insert" tab, choose a "Scatter" plot. (See example, below.)



Temperature (ºC)	Volume (L)
31.49	25
92.38	30
153.28	35
214.18	40
275.08	45
335.97	50

Your graph must include a meaningful Chart Title and Axis Titles (with units). These Chart Elements can be added to your graph by clicking on the "+" icon in the upper right corner of your graph. Your instructor may request additional Chart Elements.

To add a Trendline, right click on any data point on your graph and choose "Display Trendline" from the menu that appears. The format trendline pane will appear on the right side of your screen. Linear should be selected by default, but other types of trendlines may be tested. From this pane, you should check the box next to "Display Equation on chart." Your instructor may also ask you to check the box for "Display R-squared value on chart."

Name: ______________________________ Section: ____________________

Problem Set

1. A student performs an experiment to calculate the specific heat capacity of copper. The student experimentally finds the answer 0.340 J/g˚C. Looking up the accurate published value it is found to be 0.385 J/g˚C. Solve for the student's percent error.

$$\text{Percent error} = \left|\frac{\text{Experimental} - \text{True}}{\text{True}}\right| \times 100\ \%$$

2. Since 1965, dimes are composed of copper with 25% nickel on the outside. A Roosevelt Type dime (1946 to 1964), designed by John R Sinnockis, is composed of 90.0% silver and 10.0% copper. The composition changed when the dime cost more in silver than it was worth. A 1963 dime is weighed on ten different balances, and the mass is recorded.

Balance Number	**Mass (g) = x_m**	**$d = x_m - \bar{x}$**	**d^2**
1	2.495 g		
2	2.509 g		
3	2.507 g		
4	2.511 g		
5	2.508 g		
6	2.538 g		
7	2.512 g		
8	2.501 g		
9	2.510 g		
10	2.490 g		

a) Solve for the average value, $\bar{x}$**:** ______________________

b) Fill in the chart for all the d and d^2 values.

c) Solve for the standard deviation, s.

$$s = \sqrt{\frac{\sum d^2}{n-1}}$$

d) Solve for the range, $\bar{x} \pm 2s$.

e) Check all data points against the range. Identify values outside the range that may be unreliable and discarded:

f) Do you suspect the differing values are due to random errors or systematic errors or possibly both? Explain. How might you test your hypothesis?

g) Solve for the new average value, $\bar{x}$, removing values outside the range in part (e).

Note: Once unreliable data points are discarded the process should be repeated to recalculate $\bar{x}$, d, s, and range values. Only the recalculation of $\bar{x}$ is required for the problem set today, but you are encouraged to recalculate d, s and range on your own.

Name: ______________________________ Section: ____________________

3a) A student determines the concentration of a sodium hydroxide solution by titration with standardized KHP. S/he obtains the values: 0.190 M, 0.202 M, and 0.205 M. Should the value 0.190 M be rejected? Apply the *Q Test*. For three values Q must be greater than 0.94 to reject the number.

$$Q = \left| \frac{\text{suspect} - \text{nearest}}{\text{largest} - \text{smallest}} \right|$$

b) The student decides to repeat the experiment two more times. The five values now include: 0.190 M, 0.202 M, 0.205 M, 0.201M and 0.203M. Use the *Q Test* to see if the first value may be rejected. For five values Q must be greater than 0.64 to reject the number.

c) Solve for the average Molarity of the measurements from part b with and without the rejected number. Is there value in repeating an experiment several times?

Name: ______________________________ Section: ____________________

4. (Take home assignment). A set of solution densities as a function of weight/volume % sugar is given below. Note that weight/volume % sugar refers to how many grams of sugar per 100 mL of solution. As an example, 9.000 % means that there are 9.000 g of sugar per 100 mL of solution. Use Excel® (or similar program) to construct a density (y-axis) versus weight/volume % sugar (x-axis) plot. Add a linear fit through the Add Trendline function and display the equation and the r^2 value on your chart. Examine your r^2 value and your plot. You will notice, upon visual inspection, that there are four data points that can be considered outliers. Remove these data points, one set at a time by highlighting and then deleting the x,y values on the columns. As you delete the outliers, one data set at a time, you will see that the graph, the equation and the r^2 change accordingly. Note how the r^2 value changes. By the last deletion, you will now have an r^2 value that is generally acceptable.

   Print out two graphs, *(1) use all data points, (2) without all four outliers,* and submit to your instructor in lab next week. Graphs should conform to the five guidelines given in the introduction.

   Display the equation and r^2 value.

weight/volume % sugar	density of solution (g/mL)
0.00	0.998
2.007	1.017
3.070	1.002
4.000	1.009
5.010	1.008
6.094	1.036
6.991	1.017
8.008	1.020
9.000	1.028
10.00	1.030
11.12	1.033
12.11	1.053
13.01	1.041
15.00	1.050
16.00	1.055
17.02	1.055
18.00	1.056
19.00	1.060
21.03	1.071
23.05	1.066
24.02	1.080

Name: ________________________________ Section: ____________________

Moorpark College Chemistry Laboratory Report Rubric

(Expectations for an excellent report)

Content Area	Description
Title	Complete, appropriate title that clearly lets the reader know the topic studied.
Abstract	Clear, concise, and thorough summary of the experiment. An abstract includes a clear, complete purpose, a very brief method, major results and major discussion points/conclusions. This concise abstract generally will have four to eight sentences, approximately half a page or less.
Introduction	Clearly and thoroughly summarizes key background material and concepts pertinent to the experiment. Introduces the mathematical equations that will be necessary. Cites the sources / references that provide this background information (a minimum of two outside sources beyond the lab manual). Includes a clear and complete purpose
Method/ Materials	Outlines experimental procedure in a narrative third-person voice. Includes materials including concentrations, equipment used, a listing of safety information and waste disposal. A table is inserted when the experiment has repeating steps with varying trials and amounts. Data from experimental results do not belong here.
Results/ Calculations	Starts with a narrative sentence that introduces the data. Includes all appropriate tables, graphs, and charts with proper labels and correct information. Includes all appropriate sample calculations and has clear analysis of data with specific examples. Each graph, table or math calculation is titled or numbered and referred to in the written narrative.
Discussion/ Conclusions	Demonstrates a logical, coherent working knowledge and understanding of important experimental concepts. Forms conclusions based on the background concepts introduced in the introduction and the interpretations of results. Includes applications of and improvements in the experiment. Demonstrates accountability by providing justification for errors. Refers to the literature when appropriate.
Reference	Complete and correct citation. If the source is from a website, it must include the date the site was viewed. Samples for proper citations can be found on the Moorpark College Library website and the Chemistry Department sample formal report.
Appendix	All required supplemental components are included and complete.

Periodic Table of the Elements

I A 1	II A 2	III B 3	IV B 4	V B 5	VI B 6	VII B 7	---- 8	VIII B 9	---- 10	I B 11	II B 12	III A 13	IV A 14	V A 15	VI A 16	VII A 17	VIII A 18
1 H 1.008																	2 He 4.003
3 Li 6.941	4 Be 9.012											5 B 10.811	6 C 12.011	7 N 14.007	8 O 15.999	9 F 18.998	10 Ne 20.180
11 Na 22.990	12 Mg 24.305											13 Al 26.982	14 Si 28.086	15 P 30.974	16 S 32.066	17 Cl 35.453	18 Ar 39.948
19 K 39.098	20 Ca 40.078	21 Sc 44.956	22 Ti 47.88	23 V 50.942	24 Cr 51.996	25 Mn 54.938	26 Fe 55.847	27 Co 58.933	28 Ni 58.693	29 Cu 63.546	30 Zn 65.39	31 Ga 69.723	32 Ge 72.61	33 As 74.922	34 Se 78.96	35 Br 79.904	36 Kr 83.80
37 Rb 85.468	38 Sr 87.62	39 Y 88.906	40 Zr 91.22	41 Nb 92.906	42 Mo 95.94	43 Tc (98)	44 Ru 101.07	45 Rh 102.91	46 Pd 106.42	47 Ag 107.87	48 Cd 112.41	49 In 114.82	50 Sn 118.71	51 Sb 121.76	52 Te 127.60	53 I 126.90	54 Xe 131.29
55 Cs 132.91	56 Ba 137.33	57 La 138.91	72 Hf 178.49	73 Ta 180.95	74 W 183.84	75 Re 186.21	76 Os 190.23	77 Ir 192.22	78 Pt 195.08	79 Au 196.97	80 Hg 200.59	81 Tl 204.38	82 Pb 207.2	83 Bi 208.98	84 Po (209)	85 At (210)	86 Rn (222)
87 Fr (223)	88 Ra 226.03	89 Ac 227.03	104 Rf (261)	105 Db (262)	106 Sg (266)	107 Bh (264)	108 Hs (277)	109 Mt (268)	110 Ds (281)	111 Rg (272)	112 Cn (285)	113 Nh (284)	114 Fl (289)	115 Mc (288)	116 Lv (293)	117 Ts (294)	118 Og (294)

58 Ce 140.12	59 Pr 140.91	60 Nd 144.24	61 Pm (145)	62 Sm 150.36	63 Eu 151.96	64 Gd 157.25	65 Tb 158.92	66 Dy 162.50	67 Ho 164.93	68 Er 167.26	69 Tm 168.93	70 Yb 173.04	71 Lu 174.97
90 Th 232.04	91 Pa 231.04	92 U 238.03	93 Np 237.05	94 Pu (244)	95 Am (243)	96 Cm (247)	97 Bk (247)	98 Cf (251)	99 Es (252)	100 Fm (257)	101 Md (258)	102 No (259)	103 Lr (260)