# Valencia College
# Osceola/Lake Nona Campus

# Experimental Laboratory Manual
# for General Chemistry-II

# First Edition

# 2012

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# Table of Contents

# Experiment #1:
# Unknown Determination of a Pure Volatile Liquid by a Micro–Boiling Point

**Introduction to Vapor Pressure and Boiling Point**:
1) Liquids (along with some solids) are continuously vaporizing.
   a. Vaporization is the endothermic process by which energy is absorbed to convert a liquid into a vapor/gas.
   b. If this process occurs in a closed system, the partial pressure of the gas builds up in the space above the liquid.
2) The **vapor pressure** of a liquid is the partial pressure of the vapor over the liquid, measured at equilibrium at a given temperature.
   a. The vapor pressure of a liquid depends on its temperature.
   b. As the temperature increases, the kinetic energy of the molecular motion becomes greater and the vapor pressure increases.
   c. Liquids and solids with relatively high vapor pressures at normal temperatures are said to be **volatile.**
3) The temperature at which the vapor pressure of a liquid equals the pressure exerted on the liquid is called the **boiling point**.
   a. As the temperature of a liquid increases, the vapor pressure increases until it reaches atmospheric pressure.
   b. At this point, stable bubbles of vapor form within the liquid; this is called **boiling.**
   c. The normal boiling point is the boiling point measured at 1 atm (or 760 mm Hg).
4) To summarize, liquids with high vapor pressures have low boiling points and are considered volatile.
5) All pure liquids have a distinct boiling point (and vapor pressure) and this characteristic physical property is attributed to the intermolecular forces of the liquid.

**Introduction to Intermolecular Forces**:
1) Some liquids require more energy to be added to boil than others due to a number of attractive intermolecular forces between neighboring molecules.
   a. **Dipole–dipole interactions**: the attractive intermolecular force resulting from the tendency of polar molecules to align themselves such that the positive end of one molecule is in proximity to the negative end of another and vice versa.
$$\delta- \qquad \delta+$$
      i. **Example**: H–Cl· · · · ·H–Cl
   b. **London (dispersion) forces**: the weak attractive forces between molecules resulting from small, instantaneous dipoles that occur due to varying positions of electrons during their motion around nuclei.
      i. London forces increase with molecular weight; the larger atoms are more polarizable (or their large electron cloud can easily become distorted.)
      ii. Example:
         1. Boiling point of $CH_4$: $-161.45^{\circ}C$
         2. Boiling point of $CH_2Cl_2$: $39.8^{\circ}C$

c. **Hydrogen bonding**: the attractive force that exists between a hydrogen atom covalently bonded to a very electronegative atom and a lone pair of electrons on another small, electronegative atom.
    i. To exhibit hydrogen bonding, only nitrogen (N), oxygen (O), and fluorine (F) are electronegative enough to leave the hydrogen nucleus exposed.

    ii. Example: $CH_3OH$
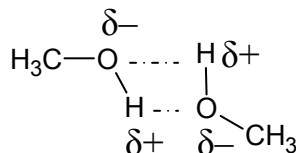
2) Two additional properties that help characterize liquids are surface tension and viscosity.
   a. **Surface tension**: the energy required to increase the surface area of a liquid by a unit amount.
     i. The International System of Units (SI) states that the proper unit for surface tension is $J/m^2$ (N/m) but commonly reported as mN/m.
     ii. A small, steel pin will float on the surface of water because water has a relatively high surface tension.
       1. The surface tension of water is $7.3 \times 10^{-2}$ $J/m^2$ or 72.80 mN/m.
   b. **Viscosity**: the resistance to flow that is exhibited by all liquids and gases.
     i. The SI unit for viscosity is kg/m•s or Pa•s.
       1. Examples:
         a. The viscosity of water is $8.90 \times 10^{-4}$ kg/m•s (Pa•s) at $25^{o}C$.
         b. The viscosity of glycerol is $1.5 \times 10^{0}$ kg/m•s (Pa•s) at $25^{o}C$.

## The Unknown Volatile Liquids:
 1) Acetone (2–propanone)
 2) Ethanol (ethyl alcohol)
 3) Isopropyl acetate (1–methylethyl ethanoate)
 4) Isopropyl alcohol (2–propanol)
 5) Methanol (methyl alcohol)
 6) Pentane

## Pre–Lab Information:
 1) In preparation for the laboratory experiment, complete "Table 1: Boiling Points and Vapor Pressures for the Unknown Volatile Liquids" in advance of the lab period and properly reference the source(s) of information.
 2) Note: You can register for a free vapor pressure calculation tool at http://www.envmodels.com/.

## The Experimental Procedure:

1) Clean and completely dry two 25 mL graduated cylinders (that are marked with 0.2 mL increments), two evaporating dishes, and one 16 x 150mm test tube.
2) Obtain 25.0 mL of an unknown volatile liquid. Record the unknown number/letter in your lab notebook.
   a. Place the liquid into one of the 25 mL graduated cylinders. Record volume in lab notebook.
3) To the other 25 mL graduated cylinder, add 25.0 mL of deionized/distilled water. Record volume in lab notebook.
4) Simultaneously and from the same height, drop a boiling bead into each graduated cylinder.
   a. Record results in your observations. Repeat at least five times to ensure reproducibility.
   b. Speculate on the viscosity of your unknown solution in comparison with water.
5) Carefully, pour your unknown solution into one clean evaporating dish without allowing the boiling beads to transfer into the dish.
6) Carefully, pour your deionized/distilled water into the other clean evaporating dish without allowing the boiling beads to transfer into the dish.
7) Gently, place a steel desk pin onto the surface of each liquid.
   a. Record results in your observations.
   b. Remove pin with a clean pair of tweezers or forceps and repeat at least five times to ensure reproducibility.
   c. Speculate on the surface tension of your unknown solution in comparison with water.
8) Carefully transfer approximately 5.0 mL of the unknown liquid into the clean and completely dried 16 x 150mm test tube.
9) Secure the test tube onto a ring stand or the metal scaffolding with a clamp.
10) Obtain a capillary tube with one open end and one closed end.
11) Rubber band the capillary tube open end down to the bulb end of a thermometer.
    a. The open end of the tube should be next to the thermometer bulb.
    b. Make certain that the rubber band does not occlude the test tube.
    c. Make sure the rubber band does not come into contact with the liquid.
12) Place the thermometer with capillary tube into the solution and secure. (See model attached.)
    a. Record the initial temperature of the unknown solution.
    b. Report observations of the liquid in the capillary tube.
13) Immerse test tube into a water bath heated on a hotplate.
14) Heat until a steady stream of bubbles can be observed from the open end of the capillary tube.
15) As soon as the steady stream of bubbles is observed, turn off heat and wait.
    a. You should notice that as the water/sample cools, the bubbles will become less frequent.
16) Record the temperature at which the liquid reenters the capillary tube.
    a. This is the first value for your boiling point range.
    b. Record all temperatures to at least the tenths place (1 decimal place.)

17) Reapply the heat (by turning on the hotplate) and record the temperature at which the first bubble comes emerges from the capillary tube.
    a. This is the second value for your boiling point range.
18) Repeat micro–boiling point procedure for a second trial using a new capillary tube and record the boiling point range.
19) If a large deviation exists between the first and second measurement, repeat for a third trial.
20) Compare the boiling point data obtained to the true values for all of the unknown liquids and identify the compound.

## Waste Disposal and Lab Clean–Up:
1) All boiling beads and steel desk pins should be cleaned, dried, and placed back into the container.
2) All water baths can be poured down the sink.
3) Extra volumes of the unknown volatile liquids should be placed in the appropriately labeled waste containers.

## The Lab – Write Up:
1) In your conclusion, discuss the important data points collected. Compare the micro–boiling point(s) of your unknown liquid to the true values for all of the known liquids and identify the compound.
2) In detail, discuss reasons why the boiling point may not have been exact.
    a. Elaborate on how the error directly affects the results.
3) Discuss the surface tension and viscosity of your unknown liquid compared to water. Approximate values for the unknown with respect to the known values for water. Discuss limitations for these tests.
4) Compare the vapor pressures for all known volatile liquids. Which chemical is the most volatile? Which is the least? How do vapor pressures relate to the boiling point?
5) What intermolecular forces (of the ones listed in the introduction) did your unknown liquid have to overcome to boil?

## Table 1: Boiling Points and Vapor Pressures for the Unknown Volatile Liquids:

| Name of Volatile Liquid | Boiling Point ($^{o}$C) | Vapor Pressure (mm Hg)* *at 20.0$^{o}$C or 293.15 K |
|---|---|---|
| Acetone (2–propanone) | | |
| Ethanol (ethyl alcohol) | | |
| Isopropyl alcohol (2–propanol) | | |
| Isopropyl acetate (1–methylethyl ethanoate) | | |
| Methanol (methyl alcohol) | | |
| Pentane | | |

References: _____

_____

_____

## Sample Data Table:

| Data Recorded/Calculated | Data Point |
|---|---|
| a) Unknown volatile liquid number/letter: | |
| | |
| b) Volume of unknown liquid in milliliters: | |
| c) Volume of deionized/distilled water in milliliters: | |
| | |
| d) Viscosity of unknown liquid in comparison with water; select from higher, lower, or equivalent | |
| | |
| e) Surface tension of unknown liquid in comparison with water; select from higher, lower, or equivalent | |
| | |
| f) Initial temperature of unknown liquid in degrees Celsius: | |
| | |
| g) Boiling point range of unknown liquid in degrees Celsius: | Trial 1: _____ <br> Trial 2: _____ <br> Trial 3: _____ |
| h) Identity of unknown volatile liquid: | |

## Model Apparatus Set – Up:



Water Fill Line

Unknown Liquid Fill Line
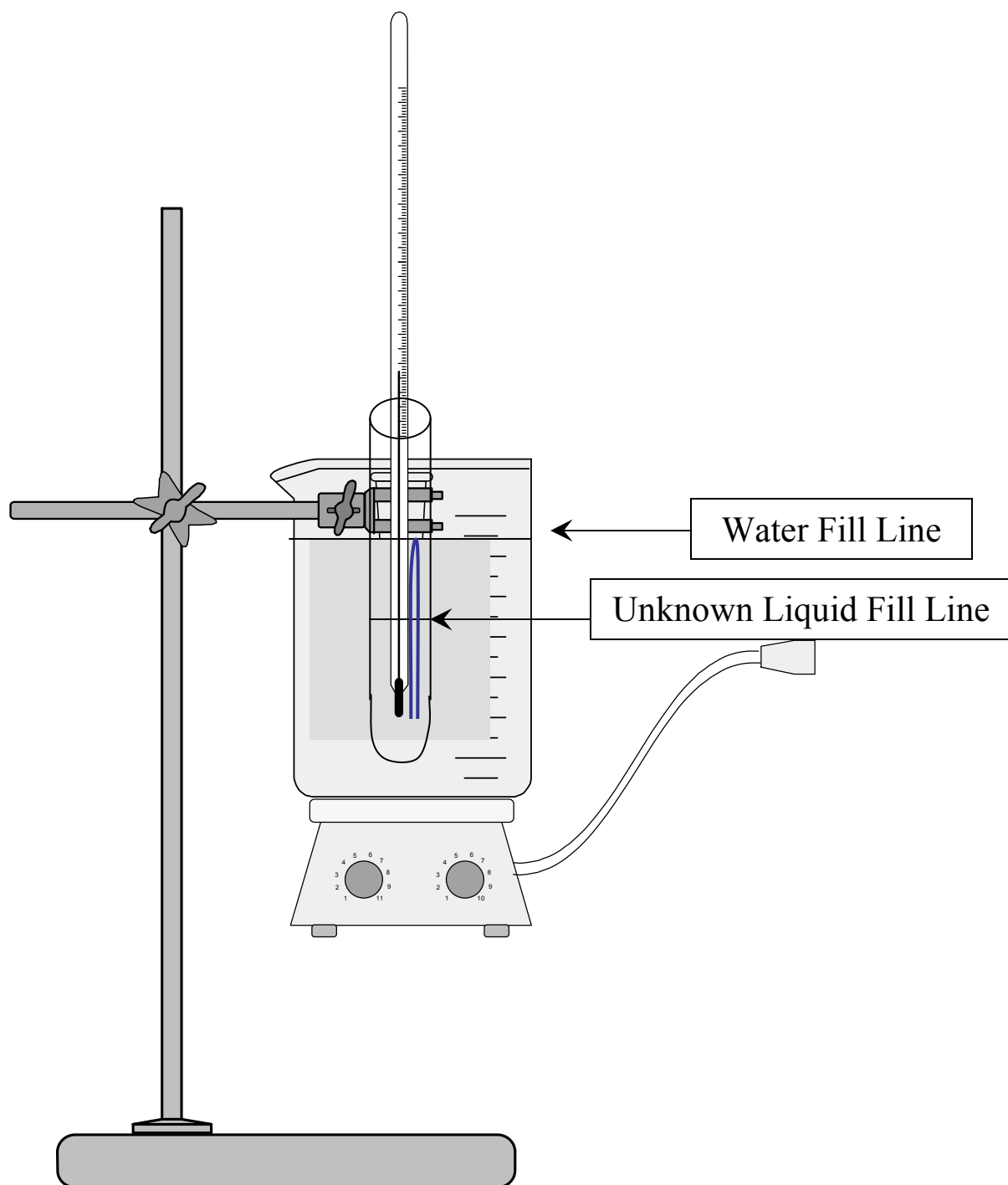
# Experiment #2:
# Determination of the Molar Mass of a Solute by Freezing – Point Depression

## Introduction to Colligative Properties:

1) Colligative properties of solutions are those properties which are dependent upon the solute concentration.
   a. These properties are independent of the nature of the solute.
2) Concentrations can be reported in several ways.
   a. Molarity ($M$) = moles of solute/liters of solution
   b. Mass percentage = mass of solute/mass of solution X 100%
   c. Molality ($m$) = moles of solute/kilograms of solvent
3) Colligative properties include the following:
   a. **Vapor pressure reduction**
      i. Vapor pressure for a nonelectrolyte, nonvolatile solute: $P_{solution} = (P^o_{solvent})(\chi_{solvent})$
      ii. Vapor pressure for a volatile solute: $P_{solution} = (P^o_{solvent})(\chi_{solvent}) + (P^o_{solute})(\chi_{solute})$
      iii. Vapor pressure reduction: $\Delta P_{solv} = (P^o_{solvent})(\chi_{solute})$
   b. **Freezing point depression**
      i. Formula for nonelectrolytic solutes: $\Delta T_f = (K_f)(c_m)$
      ii. Formula for electrolytic solutes: $\Delta T_f = i(K_f)(c_m)$
   c. **Boiling point elevation**
      i. Formula for nonelectrolytic solutes: $\Delta T_b = (K_b)(c_m)$
      ii. Formula for electrolytic solutes: $\Delta T_b = i(K_b)(c_m)$
   d. **Osmosis**
      i. Formula for nonelectrolytic solutes: $\Pi = MRT$
      ii. Formula for electrolytic solutes: $\Pi = iMRT$
4) The variable $i$, or the van't Hoff factor, expresses the number of particles (or ions) that the substance would produce when dissolving.
   a. Ionic solutes generally have a van't Hoff factor that is greater than 1.
      i. Example: $CaCl_2$, $i = 3$ ($Ca^{2+}$ and 2 $Cl^-$)
   b. Molecular solutes often have a van't Hoff factor that is equal to 1 because they do not dissociate upon dissolving.
      i. Example: $C_6H_{12}O_6$, $i = 1$
5) Colligative properties can be used to determine the molar mass for unknown solutes or assess purity of known substances.

## Introduction to Freezing Point Depression:

1) Cyclohexane is a common nonpolar solvent that occurs naturally in petroleum.
   a. It is an extremely volatile liquid and is used as the solvent in paint and varnish remover.
   b. It has a distinct freezing point of $6.47^oC$ and a freezing point depression constant ($K_f$) of $20.5^oC/m$.

2) In order to determine the freezing point depression ($\Delta T_f$), the freezing point of pure cyclohexane and the freezing point of a solution of the solute in cyclohexane must be determined.

3) Cooling curves, which measure temperature as a function of time, will be constructed for the pure sample and for the solution.

    a. The freezing point ($t_f$) will appear as a plateau in the middle of the cooling curve for the pure cyclohexane. See Graph 1.

    b. The freezing point ($t_f$) will appear as the intersection of two straight lines of the cooling curve for the cyclohexane solution. See Graph 2.

    c. To obtain a smooth cooling curve, the solvent must be adequately stirred.

    d. Without stirring, the cooling curve appears as "steps" that result in inaccurate data.

## Graph 1: Temperature versus time for pure cyclohexane
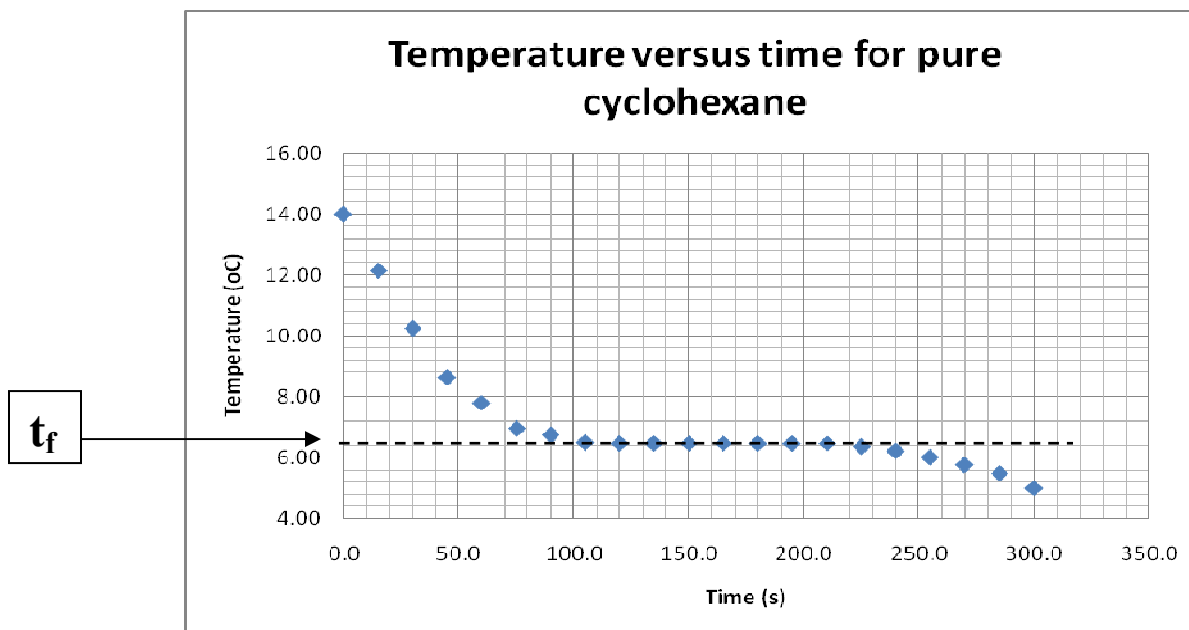
## Graph 2: Temperature versus time for cyclohexane solution
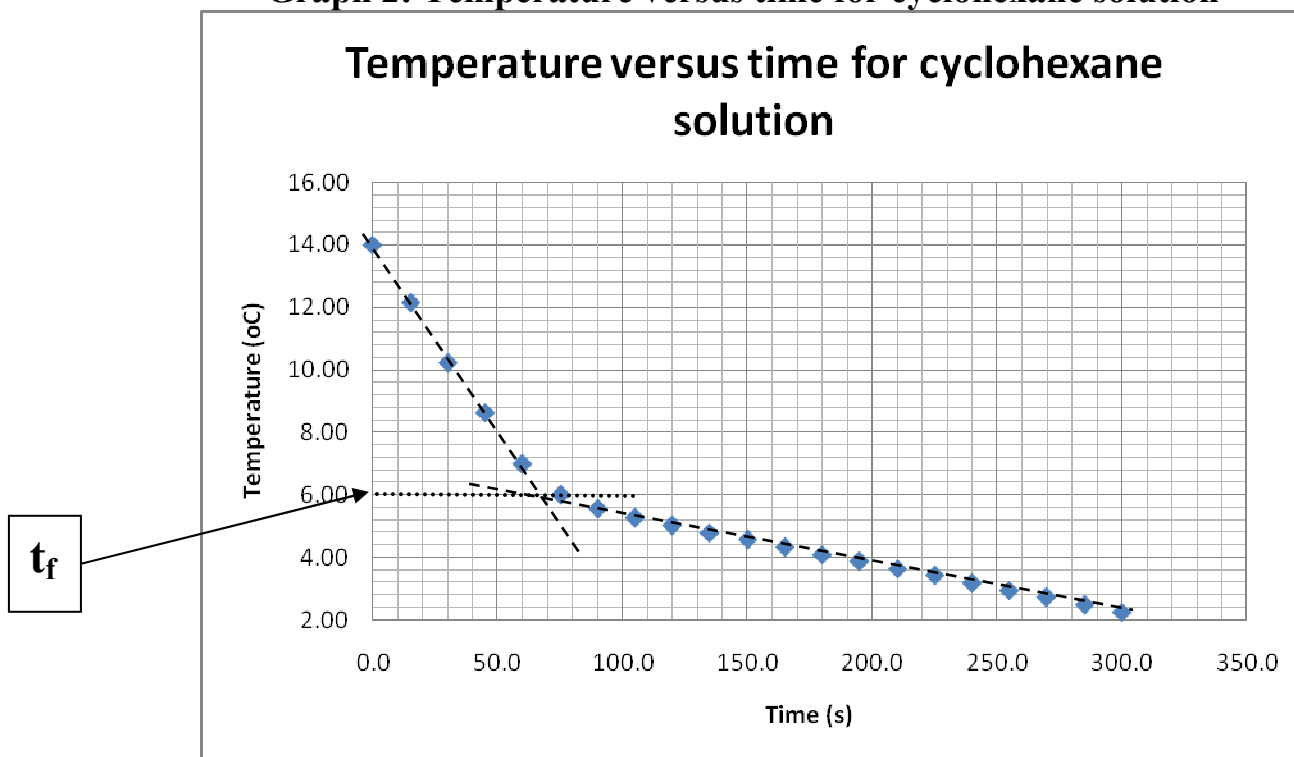


## The Possible Solutes:

1) Biphenyl
2) Camphor (or *DL*–Camphor)
3) 1,4–Dichlorobenzene (or *para*–Dichlorobenzene)
4) Diphenylamine
5) Menthol
6) Naphthalene
7) 2–Nitrobiphenyl
8) Thymol

## Part 1

### Procedure for Measuring the Freezing Point of Pure Cyclohexane:

1) Obtain a clean and completely dry 25 mL volumetric flask, a small magnetic stir bar, and a long mercury thermometer*.
   a. *An alternative set–up can be implemented using temperature probes in place of thermometers.
   b. If small magnetic stir bars are unavailable, a copper wire can be placed used for manual stirring.
2) Pipet exactly 20.0 mL of cyclohexane into the volumetric flask.
   a. Never pipet by mouth; use a bulb or pipetting device/pump.
   b. Calculate and record the mass of cyclohexane in your lab notebook. Hint: Obtain the density of cyclohexane.
3) Carefully, insert a small magnetic stir bar into the cyclohexane.

a. Ensure no solvent splashes out!
b. If a small stir bar is not available, use a copper wire.
4) Carefully, insert the thermometer into the volumetric flask and ensure the bulb is completely submerged in the cyclohexane.
5) Clamp the volumetric flask to a ring stand or to the lab scaffolding.
6) Obtain a hot plate with stirring capabilities.
a. Place beneath volumetric flask and turn on stir knob to ensure that the stir bar circulates the cyclohexane.
b. You will NOT need the heating function for the hot plate.
7) In a 400–600 mL beaker, create an ice–water bath.
a. Make sure you have a large volume of ice in the water.
b. You will need to replenish the ice throughout the experiment so fill a separate beaker with ice and have it readily available.
8) Place the ice–water bath onto the hot plate.
9) Immerse the volumetric flask into the ice–water bath.
a. Ensure that the stir bar is actively stirring the cyclohexane.
b. Also, ensure that no water gets into the volumetric flask.
10) Once the temperature has dropped to about $14^{o}C$, begin recording the temperature to the nearest $0.01^{o}C$ (or $0.1^{o}C$ depending on the sensitivity of the instrument) every 15 seconds.
a. In general, approximately up to ten minutes is required for this recording step.
11) After the cyclohexane has frozen and the temperature has begun to decline below the freezing point plateau, remove the volumetric flask from the ice–water bath.
12) Allow the cyclohexane to warm up to room temperature and melt completely.
a. The volumetric flask can be cupped in a gloved hand to warm up quickly.
13) Repeat steps 9 – 12 for a second trial.
14) Construct a temperature as a function of time curve to adequately calculate the mean freezing point for both trials.
a. Ensure major and minor gridlines are turned on.
15) Calculate the mean freezing point.
a. The freezing point ($t_f$) will appear as a plateau in the middle of the cooling curve for the pure cyclohexane.
16) This cyclohexane will be used for the next part of the experiment; do not discard.

## Part 2
**Procedure for Measuring the Freezing Point of a Cyclohexane Solution**:
1) You will be assigned one known solute; record this information in your lab notebook.
2) Obtain two pieces of waxed weighing paper.
3) Weigh out approximately 0.25 g of solute onto the first weighing paper using an analytical balance.
a. Record the exact mass of solute in the lab notebook.
b. Label weighing paper as #1 or with the mass with a wax pencil.
c. DO NOT allow the mass of any solute to touch the plate of the balance.
d. Never add solute to the weighing paper INSIDE of the balance; do this outside the balance and then place onto the balance.

4) Weigh out approximately 0.10 g of solute onto the second weigh paper using the same analytical balance.
   a. Record the exact mass of solute in the lab notebook.
   b. Label weighing paper as #2 or with the mass with a wax pencil.
5) Transfer the first solute sample (weighing paper #1) into the pure cyclohexane sample.
6) Stir until all solute has dissolved.
7) After the solute has dissolved, place the ice–water bath onto the hot plate.
   a. You may have to replenish the ice in the bath.
8) Immerse the volumetric flask into the ice–water bath.
   a. Ensure that the stir bar is actively stirring the cyclohexane solution.
9) Once the temperature has dropped to about 14°C, begin recording the temperature to the nearest 0.01°C (or 0.1°C depending on the sensitivity of the instrument) every 15 seconds.
10) Continue collecting data until the solution is completely frozen.
11) After the cyclohexane solution has frozen, remove the volumetric flask from the ice–water bath and allow the solution to melt completely.
    a. The volumetric flask can be cupped in a gloved hand to warm up quickly.
12) As the solution is melting, check data to ensure that the temperature has fallen smoothly. If a stair–step effect can be observed, repeat steps 8 – 11.
13) Add the second sample of solute to the solution (weigh paper #2) and allow to dissolve completely.
    a. Record data under the "Sample #1 + #2" section since you are using a combined mass of solute.
14) Repeat steps 8 – 11 with the new solution.
15) Once complete, construct a temperature as a function of time curve to adequately calculate the freezing point for the solution for both trials.
    a. The freezing point (t$_f$) will appear as the intersection of two straight lines of the cooling curve for the cyclohexane solution.
    b. Ensure major and minor gridlines are turned on.
16) Calculate the freezing point depression ($\Delta T_f$) and the molar mass (M$_m$) of the solute for each solution.
17) Calculate the average molar mass and compare to the true molar mass of your known solute.

## Waste Disposal and Lab Clean–Up:
1) All ice water baths can be discarded down the drain UNLESS contaminated with cyclohexane.
2) All cyclohexane solutions should be place into the appropriately labeled waste container.
   a. DO NOT discard these solutions down the drain!

## The Lab Write – Up:
1) Determine all values in your data table and discuss relevant data points in your conclusion.
2) Determine the molar mass of your solute and compare the experimental mean molar mass to that of the true molar mass.
3) Calculate the percent error and discuss reasons for possible deviations in detail.

    a. Percent error = [(true value – experimental value)/true value] * 100%

    b. Percent error cannot be a negative value.  Take the absolute value of the number if necessary.

6) What would happen to the calculated molar mass if some cyclohexane was allowed to evaporate?

7) What would happen to the calculated molar mass if the cyclohexane was contaminated with another solute?

8) If the solutes assigned were unknown, determination of the molar mass via freezing–point depression could determine the identity.  What other tests could have been performed on the unknown solute to determine identity?

# Sample Data Table:

| Data Recorded/Calculated | Data Point(s) |
|---|---|
| a) Solute name: | |
| | |
| b) Volume of cyclohexane used in milliliters: | |
| c) Mass of cyclohexane used in grams: | |
| d) Mass of cyclohexane used in kilograms: | |
| | |
| e) Freezing point of pure cyclohexane via graph in degrees Celsius: | Trial 1: _____<br>Trial 2: _____ |
| f) Mean freezing point of pure cyclohexane in degrees Celsius ([Trial 1 + Trial 2]/2): | |
| | |
| g) Mass of solute in sample #1 in grams: | |
| h) Mass of solute in sample #2 in grams: | |
| i) Combined mass of solute (sample #1 + #2) in grams (g + h): | |
| | |
| j) Freezing point of solution via graph for sample #1 in degrees Celsius: | |
| k) Freezing point depression ($\Delta T_f$) of solution for sample #1 in degrees Celsius (f–j): | |
| | |
| $\ell$) Molality ($c_m$) of solute in cyclohexane solution for sample #1 in mol/kg[†]: | |
| m) Moles of solute in cyclohexane solution for sample #1 in mol (d* $\ell$): | |
| n) Molar mass ($M_m$) of solute for sample #1 in grams per mole (g/m): | |
| | |
| o) Freezing point of solution via graph for sample #1 + #2 in degrees Celsius: | |
| p) Freezing point depression ($\Delta T_f$) of solution for sample #1 + #2 in degrees Celsius (f–o): | |
| | |
| q) Molality ($c_m$) of solute in cyclohexane solution for sample #1 + #2 in mol/kg[†]: | |
| r) Moles of solute in cyclohexane solution for sample #1+2 (d*q): | |
| s) Molar mass ($M_m$) of solute for sample #1 + #2 in grams per mole (i/r): | |
| | |
| t) Mean (average) molar mass ($M_m$) of solute in grams per mole ([n+s]/2): | |
| u) True molar mass ($M_m$) of solute in grams per mole: | |
| v) Percent error from true molar mass in percent: | |

[†]Use the following formula and solve for molality: $\Delta T_f = i(K_f)(c_m)$

# Freezing Point Data of Pure Cyclohexane:

| Trial #1 | | Trial #2 | |
|---|---|---|---|
| Time (s) | Temp. ($^o$C) | Time (s) | Temp. ($^o$C) |
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# Freezing Point Data of Cyclohexane Solution:
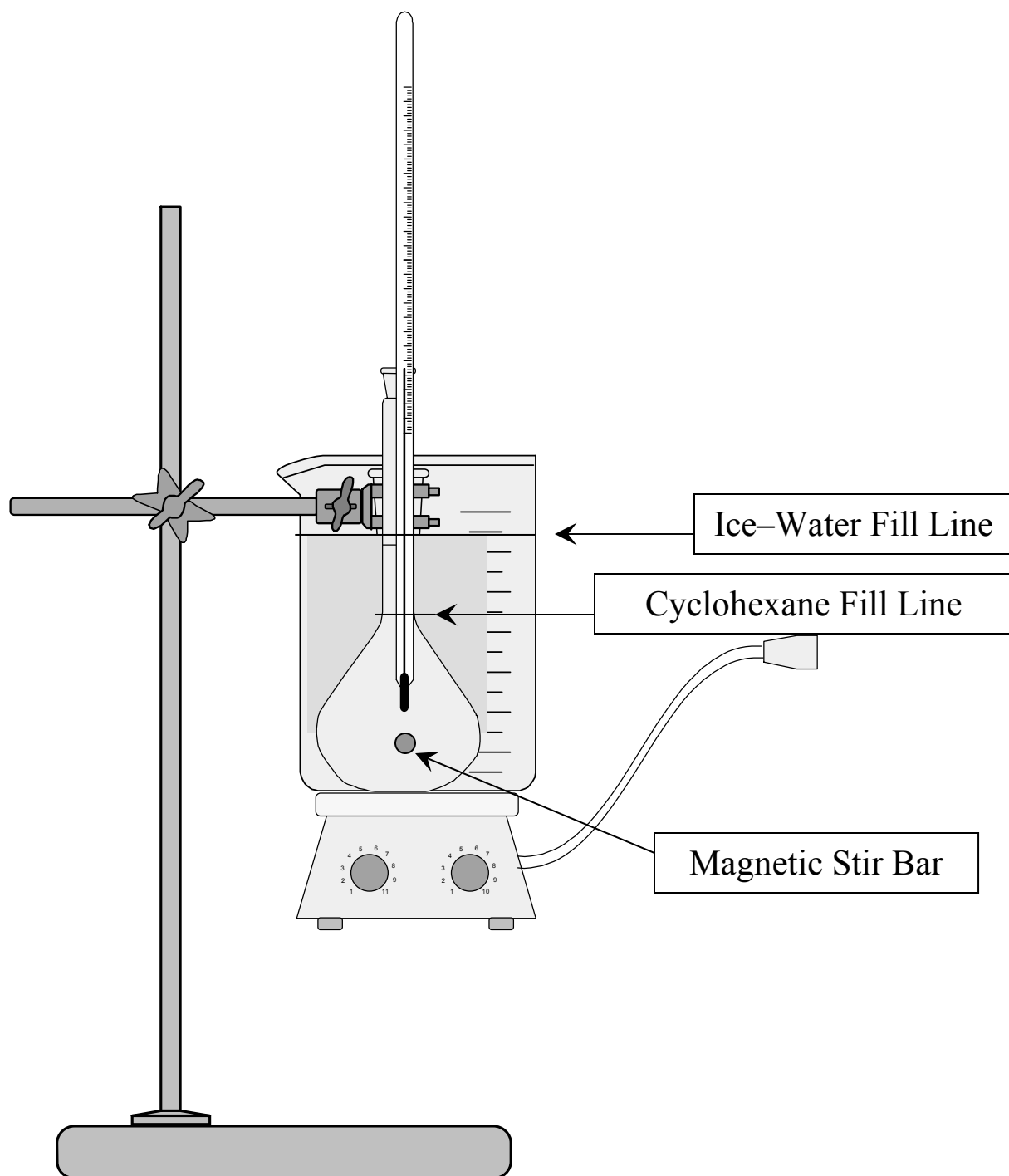
| Sample #1 | | Sample #1 + #2 | |
|---|---|---|---|
| Time (s) | Temp. ($^o$C) | Time (s) | Temp. ($^o$C) |
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## Model Apparatus Set – Up:



Ice–Water Fill Line

Cyclohexane Fill Line

Magnetic Stir Bar

## Introduction to Chemical Kinetics:
1) Chemical reactions require varying lengths of time for completion.
   a. This reaction rate depends on the characteristics of the reactants and products and the conditions under which the reaction is run.
2) **Chemical kinetics** is the study of reaction rates, how reaction rates change under varying conditions, and what molecular events occur during the overall reaction.
   a. The following variables affect the reaction rate:
      i. The concentration of the reactants
      ii. The concentration of the catalyst
      iii. Temperature of the reaction mixture
      iv. Surface area of a solid reactant or catalyst

## The Factors Affecting Reaction Rates:
1) **The concentration of reactants**:
   a. Often, the rate of a reaction <u>increases</u> when the concentration of a reactant is increased.
   b. Increasing the population of reactants increases the likelihood of a successful "collision" or interaction between reactants.
   c. In some reactions, however, the rate is unaffected by the concentration of a particular reactant, as long as it is present at some concentration.
2) **The concentration of the catalyst:**
   a. A catalyst is a substance that increases the rate of a reaction without being consumed in the overall reaction.
   b. The catalyst generally does not appear in the overall balanced chemical equation (although its presence may be indicated by writing its formula over the arrow).
3) **The temperature of the reaction mixture:**
   a. Commonly, the rate of the reaction increases as the temperature increases.
   b. A good "rule of thumb" is that reactions approximately double in rate with a 10 $^{\circ}$C rise in temperature.
      i. Note: This is only a generalization and is not applicable for all reactions!
   c. For reactions in solution in a fixed volume, as temperature increases, the kinetic energy of the reactants also increase causing more "collisions".
4) **The surface area of a solid reactant or catalyst:**
   a. Because the reaction occurs at the surface of the solid, the rate increases with increasing surface area.
   b. Examples:
      i. Wood combusts (burns) faster if the pieces are smaller.
      ii. Granular calcium carbonate reacts faster with hydrochloric acid than large pieces of marble or limestone.
         1. $CaCO_3(s) + 2\ HCl(aq) \rightarrow CaCl_2(aq) + 2\ H_2O(l) + CO_2(g)$

## Introduction to Reaction Rates:

1) The reaction rate is the increase in molar concentration of a product of a reaction per unit time.
   a. It can also be expressed as the **decrease** in molar concentration of a **reactant** per unit time.
2) A **rate law** is an equation that relates the rate of a reaction to the concentration of reactants (and catalyst) raised to various powers.
   a. Example reaction: $2NO_2(g) + F_2(g) \rightarrow 2NO_2F(g)$
      i. The rate law: Rate $= k[NO_2][F_2]$
      ii. Since the rate is directly proportional to the concentrations of $NO_2$ and $F_2$, the exponents are one for each (and are not shown).
         1. When the concentration of nitrogen dioxide is doubled, the reaction rate doubles.
         2. Doubling the concentration of fluorine also doubles the rate of the reaction.
   b. The following chart shows how the rate will change for certain reaction orders as the concentration of a reactant is doubled:

| Reaction Order | Rate Change |
|---|---|
| −1 | Rate decreases by ½ |
| 0 | Rate does not change |
| 1 | Rate doubles |
| 2 | Rate quadruples |

## Introduction to the Reactions:

1) **Part 1:** The first reaction is between sodium thiosulfate and hydrochloric acid to produce colloidal sulfur and salt water.
   a. The balanced equation: $Na_2S_2O_3(aq) + 2\ HCl(aq) \rightarrow 2\ NaCl(aq) + H_2O(l) + S(s) + SO_2(g)$.
   b. The concentration of both reactants will be changed individually to determine the rate law.
2) **Part 2:** The second reaction is the oxidation of glucose by potassium permanganate to carbon dioxide and water.
   a. The balanced equation: $3\ KMnO_4(aq) + C_6H_{12}O_6(aq) \rightarrow 3\ Mn^{2+}(aq) + 3\ K^+(aq) + 6\ CO_2(g) + 6\ H_2O(l)$
   a. The reaction rate will be determined by analyzing this reaction under different temperatures.
   b. Sulfuric acid is used as a catalyst for this reaction.

## An Introduction to the Graphs:

1) For part 1, there will be 2 graphs to construct.
   a. Graph #1: Plot of the thiosulfate concentration ($M$) versus the rate of the reaction ($s^{-1}$).
   b. Graph #2: Plot the hydrochloric acid concentration ($M$) versus the rate of the reaction ($s^{-1}$).

2) For part 2, there will be 1 graph.
   a. Graph #1: Plot of the temperature ($^{\circ}$C) versus the rate (s$^{-1}$).

# First order plot

*Notice: At a concentration of 4.0 M, the rate is 0.0064 s$^{-1}$ and when the concentration is doubled to 8.0 M, the rate increases to 0.0114 s$^{-1}$. This is 1.8 times the rate which is approximately doubled.*

**Concentration of _____ Versus Rate**
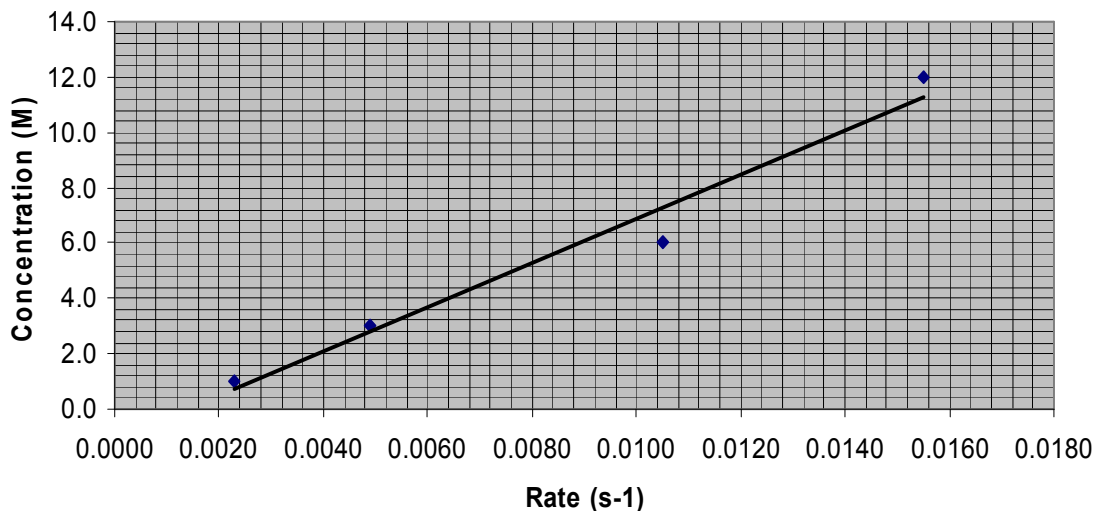


# Second order plot

*Notice: At a concentration of 4.0 M, the rate is 30 x 10$^{-4}$ s$^{-1}$ and when the concentration is doubled to 8.0 M, the rate increases to 120 x 10$^{-4}$ s$^{-1}$. This is a 4–fold increase which is second order.*

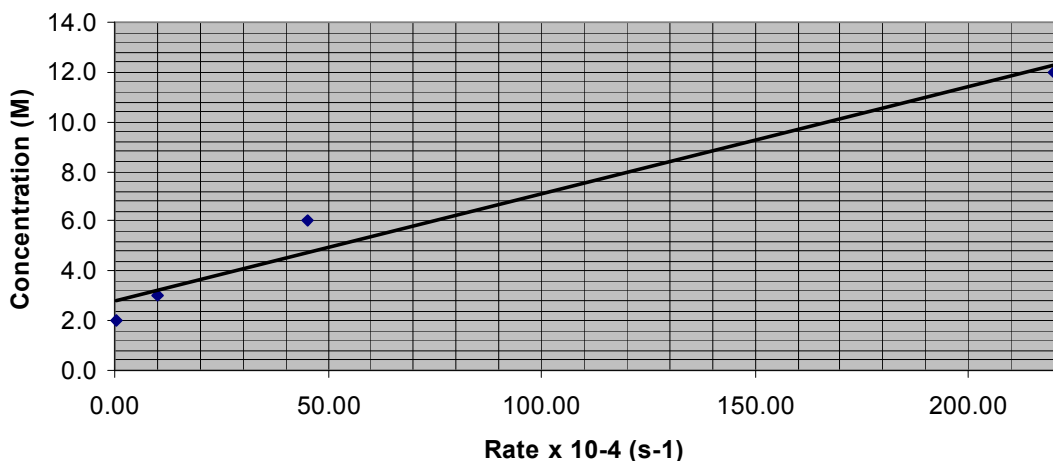**Concentration of _____ Versus Rate**

## Zero order plot

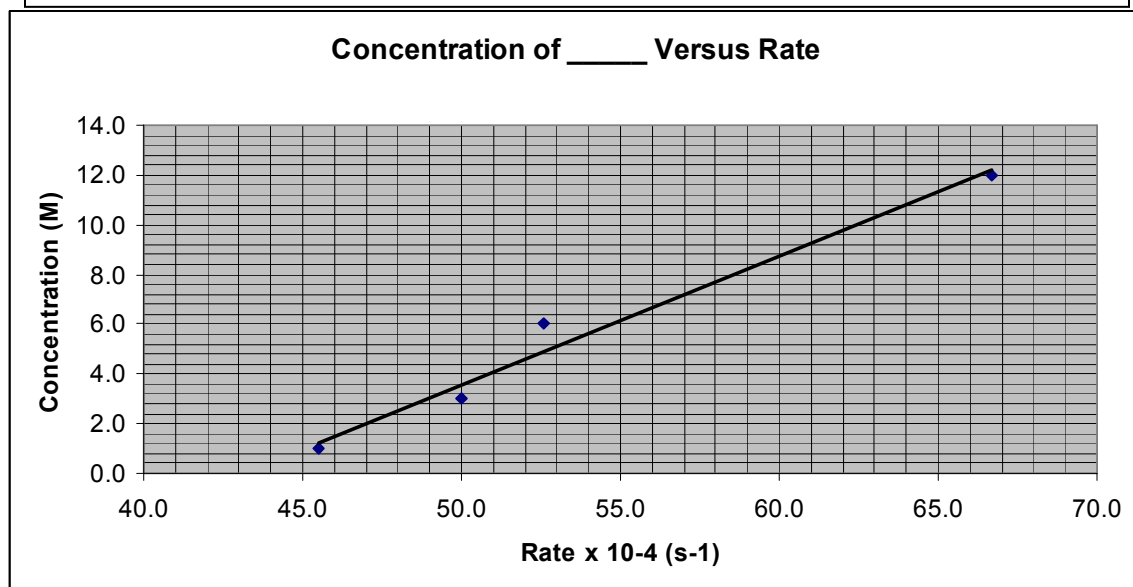*Notice: At a concentration of 2.0 M, the rate is 47 x $10^{-4}$ $s^{-1}$ and when the concentration is doubled to 4.0 M, the rate increases to 51 x $10^{-4}$ $s^{-1}$. This is a rate change of 1.09 times the original rate which is essentially no change.*

**Concentration of _____ Versus Rate**



**Part 1: Experimental Procedure for Determining the Rate Law from the Sodium Thiosulfate Reaction**:

1) Obtain 8 clean and completely dried 100–250 mL beakers, a 100.0 mL graduated cylinder, a 50.0 mL graduated cylinder, a 10.0 mL graduated cylinder, and a stirring hotplate with a stir bar.
2) Using a wax pencil, mark the numbers 1–8 on the beakers for identification purposes.
    a. Try to make the eight beakers all be the same size, if possible.
3) Measure out 90.0 mL of 0.15 *M* sodium thiosulfate ($Na_2S_2O_3$) solution.*
    a. *If not already prepared, dissolve 23.7 g $Na_2S_2O_3$ in enough deionized water to produce 1.0 L of solution.
4) Add the amounts of sodium thiosulfate solution and deionized/distilled water to the first five beakers as indicated in the following table. Mix thoroughly.

| Beaker Number | Volume of 0.15 *M* $Na_2S_2O_3$ (mL) | Volume of Deionized Water (mL) |
|---|---|---|
| 1 | 25.0 | 0.0 |
| 2 | 20.0 | 5.0 |
| 3 | 15.0 | 10.0 |
| 4 | 10.0 | 15.0 |
| 5 | 5.0 | 20.0 |

5) Using a 50.0 mL graduated cylinder, measure out 30.0 mL of a 6.0 *M* HCl solution.

      a. Place the HCl solution into a small beaker and label with a wax pencil.
      b. Wear goggles, gloves, and appropriate attire when handling strong acids.
6) Place the first beaker (beaker number 1) on the stirring hotplate, insert the magnetic stir bar, and set to a moderate stir.
7) Make a small "X" on a white sheet of paper with a pencil and place under the first beaker.
8) Using a 10.0 mL graduated cylinder, measure out 5.0 mL of 6.0 $M$ HCl solution and transfer into the first beaker.
      a. As soon as the acid touches the sodium thiosulfate solution, immediately start timing with a stop watch.
9) Cease timing when the "X" is no longer visible through the solution and record this time (in seconds) in the data table.
10) Calculate the rate of the reaction by taking the inverse of the time recorded.
11) Repeat steps 7 – 11 with the remaining beakers.
      a. Ensure that you rinse off and completely dry stir bar before proceeding to the next beaker.
12) Obtain an additional 75.0 mL of 0.15 $M$ sodium thiosulfate solution.
13) Place 25.0 mL of the sodium thiosulfate solution into beakers 6–8.
      a. Do NOT add deionized/distilled water to any of the beakers.
      b. Using the same procedure discussed previously, perform the experiment under the conditions shown in the table below:

| Beaker Number | Volume of 0.15 $M$ $Na_2S_2O_3$ (mL) | Volume (mL) & Concentration (M) of HCl |
|---|---|---|
| 6 | 25.0 | 5.0 mL 1.0 $M$ HCl |
| 7 | 25.0 | 5.0 mL 3.0 $M$ HCl |
| 8 | 25.0 | 5.0 mL 12.0 $M$ HCl |

14) As soon as the acid touches the sodium thiosulfate solution, immediately start timing with a stop watch and cease timing when the "X" is no longer visible through the solution and record this time (in seconds) in the data table.
15) Calculate the rate of the reaction by taking the inverse of the times recorded.
16) After completion, construct the following graphs:
      a. Graph #1: Plot of the thiosulfate concentration ($M$) versus the rate of the reaction ($s^{-1}$).
      b. Graph #2: Plot the hydrochloric acid concentration ($M$) versus the rate of the reaction ($s^{-1}$).
      c. Note: The concentration should go on the y–axis and the rate should be placed on the x–axis.
      d. Using linear regression, produce the best fit straight line.
17) Determine the rate equation using the data obtained from the two graphs constructed.

## Part 2: Experimental Procedure for the Oxidation of Glucose by Potassium Permanganate:

1) You will be assigned one or more of the temperature ranges in the scheme below. The entire class must share this information in order to have enough data construct the graph.
2) Obtain a 250 mL Erlenmeyer flask, a 100.0 mL graduated cylinder, a 600 mL beaker, a ring stand, a 25 mL graduated cylinder, a 2.0 mL pipette with pipetting device/pump, a 10 mL graduated cylinder, a thermometer, a ring stand, a clamp, and a stirring hotplate with a stir bar.
3) Measure out 75.0 mL of deionized/distilled water using a 100.0 mL graduated cylinder and place into a 250 mL Erlenmeyer flask.
   a. With a wax/grease pencil, label as "reaction flask."
4) Next, using a 25.0 mL graduated cylinder, carefully measure out 25.0 mL of 2.0 $M$ sulfuric acid and place into the reaction flask.
5) Using a 2.0 mL pipette with a pipetting device/pump, pipet exactly 4.0 mL of the 0.10 $M$ potassium permanganate solution into the reaction flask.
   a. Ensure pipette is thoroughly cleaned after use!
6) Using a 10.0 mL graduated cylinder, measure out 5.0 mL of 2% glucose solution but **do not** add to the flask. It will be added later when a stable temperature range of the reaction flask is achieved.
7) Add an appropriate amount of water to the 600 mL beaker and place onto the hotplate. Turn on hotplate to achieve designated temperature range.
8) Immerse the reaction flask into the water bath and clamp to a ring stand.
9) Insert stir bar into the reaction flask and allow for a moderate stirring.
10) Use a thermometer to achieve the temperature range assigned.
11) Once a reasonably stable temperature range has been achieved and has been recorded, add 5.0 mL of 2% glucose solution and start the timer.
    a. Ensure that the temperature is stable ($\pm2.0^{\circ}$C) as the reaction proceeds.
    b. Do not allow the sample to boil.
12) Record the time it takes for the purple/pink color to disappear.
13) Repeat steps 1–10 with the following temperature schemes as indicated by your instructor and share results with class:

| Beaker Number | Volume of 2.0 $M$ H$_2$SO$_4$ (mL) | Volume of 0.10 $M$ KMnO$_4$ (mL) | Volume of 2% glucose solution (mL) | Temperature Range to Achieve ($^{\circ}$C) |
|---|---|---|---|---|
| 1 | 25.0 | 4.0 | 5.0 | 50 – 60 |
| 2 | 25.0 | 4.0 | 5.0 | 60 – 70 |
| 3 | 25.0 | 4.0 | 5.0 | 70 – 80 |
| 4 | 25.0 | 4.0 | 5.0 | 80 – 90 |
| 5 | 25.0 | 4.0 | 5.0 | 90 – 99 |

14) After completion, construct a graph of the temperature ($^{o}C$) versus the rate ($s^{-1}$).
   a. The temperature should go on the y–axis and the rate should be placed on the x–axis.
   b. Using linear regression, produce the best fit straight line.


## Waste Disposal and Lab Clean–Up:
   1) Hot water baths can be discarded down the sink.
   2) Place all acidic thiosulfate solutions into the appropriately labeled waste container under the fume hood.
   3) Place all permanganate solutions into the appropriately labeled waste container.


## The Lab Write – Up:
   1) Determine all values in your data table and discuss relevant data in your conclusion.
   2) For part 1, after constructing Graph #1: Plot of the thiosulfate concentration ($M$) versus the rate of the reaction ($s^{-1}$), discuss the reaction order with respect to this reactant.
   3) For part 1, after constructing Graph #2: Plot the hydrochloric acid concentration ($M$) versus the rate of the reaction ($s^{-1}$), discuss the reaction order with respect to this reactant.
   4) For part 1, determine the rate equation for the thiosulfate experiment based on both Graph #1 and Graph #2 and discuss in your conclusion. Explain using graphical data.
   5) For part 2, after constructing a graph of the temperature ($^{o}C$) versus the rate ($s^{-1}$), make a **quantitative** statement about how the rate of glucose oxidation varies with the temperature. Compare to the general "rule of thumb" regarding the reaction rate as temperature is increased.
   6) Discuss all sources of error during this experiment and how they directly affected the data.

# Sample Data Tables:

**Part 1(a):** Using 5.0 mL of 6.0 $M$ HCl for each

| Beaker Number | Volume of 0.15 $M$ $Na_2S_2O_3$ (mL) | Volume of Deionized Water (mL) | Resultant Molarity of $Na_2S_2O_3$ ($M$) *y–axis* | Time (s) | Rate ($s^{-1}$) *x–axis* |
|---|---|---|---|---|---|
| 1 | 25.0 | 0.0 | 0.15 $M$ | | |
| 2 | 20.0 | 5.0 | | | |
| 3 | 15.0 | 10.0 | | | |
| 4 | 10.0 | 15.0 | | | |
| 5 | 5.0 | 20.0 | | | |

Reaction order with respect to $Na_2S_2O_3$*: _____

*Select from zero, first, or second

**Part 1(b):** Using 25.0 mL of 0.15 $M$ of $Na_2S_2O_3$ for each

| Beaker Number | Volume of 0.15 $M$ $Na_2S_2O_3$ (mL) | Volume (mL) & Conc. ($M$) of HCl *y–axis* | Time (s) | Rate ($s^{-1}$) *x–axis* |
|---|---|---|---|---|
| 1 (see above) | 25.0 | 5.0 mL 6.0 $M$ HCl | | |
| 6 | 25.0 | 5.0 mL 1.0 $M$ HCl | | |
| 7 | 25.0 | 5.0 mL 3.0 $M$ HCl | | |
| 8 | 25.0 | 5.0 mL 12.0 $M$ HCl | | |

Reaction order with respect to HCl*: _____

*Select from zero, first, or second

The Overall Rate Equation: _____

# Sample Data Tables (Continued):

## Part 2: Oxidation of Glucose by Potassium Permanganate:

| Beaker No. | Volume of 2.0 $M$ $H_2SO_4$ (mL) | Volume of 0.10 $M$ $KMnO_4$ (mL) | Volume of 2% glucose solution (mL) | Temperature Range to Achieve ($^oC$) | Actual Temp. ($^oC$) *y–axis* | Time (s) | Rate ($s^{-1}$) *x–axis* |
|---|---|---|---|---|---|---|---|
| 1 | 25.0 | 4.0 | 5.0 | 50 – 60 | | | |
| 2 | 25.0 | 4.0 | 5.0 | 60 – 70 | | | |
| 3 | 25.0 | 4.0 | 5.0 | 70 – 80 | | | |
| 4 | 25.0 | 4.0 | 5.0 | 80 – 90 | | | |
| 5 | 25.0 | 4.0 | 5.0 | 90 – 99 | | | |

# Experiment #4:
# The Determination of an Equilibrium Constant for a Fischer Esterification Reaction

## Introduction to the Ester:

1) The ester functional group has this generic structure:

$$R\!-\!\overset{\displaystyle\overset{O}{\|}}{C}\!-\!O\!-\!R_1$$

2) Most low molecular weight esters have a pleasant odor that comprises the major flavors or fragrances of fruits and other pleasing aromatics called "essential oils."
   a. Esters are used commercially in artificial flavors or fragrances.
3) Esters are also main components in some pheromones which elicit a social response in same species animals.

## The Esterification Process:

1) Esters can be prepared in a multitude of ways.
2) The classic ester synthesis is via a Fischer esterification by which a carboxylic acid reacts with an alcohol in the presence of one a of the following catalysts:
   a. A concentrated acid such as sulfuric acid
   b. An "alcohol activator" such as *p*–toluenesulfonic acid
   c. The acidic form of an ion exchange resin such as polymeric sulfonic acids
3) The Fischer esterification process requires a long equilibrium based reflux.
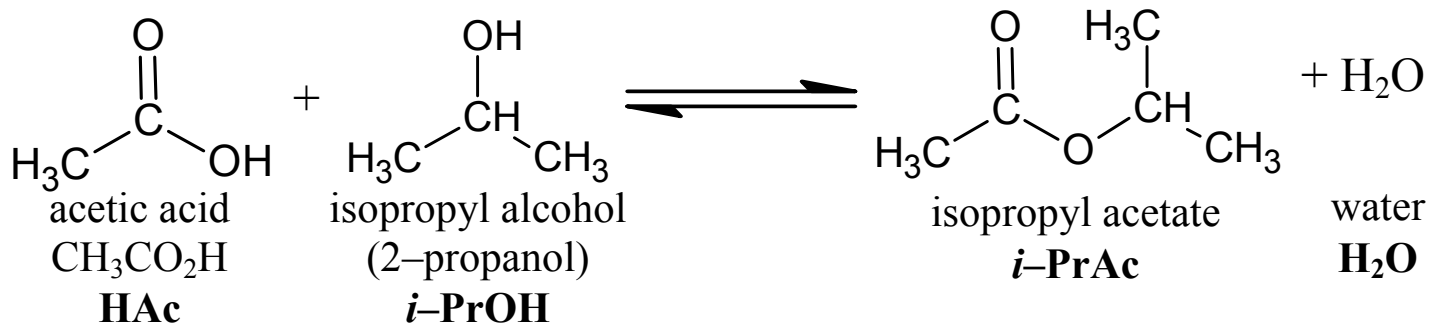   a. By manipulating Le Châtelier's principle, the reaction equilibrium can be shifted toward product formation.

## Introduction to the Equilibrium Constant:

1) A dynamic equilibrium consists of a forward reaction, in which substances react to give products, and a reverse reaction, in which products react to give the original reactants.
   a. **Chemical equilibrium** is the state reached by a reaction mixture when the rates of the forward and reverse reactions have become equal.
2) Every reversible system has its own "position of equilibrium" under any given set of conditions.
   a. The ratio of products produced to unreacted reactants for any given reversible reaction remains constant under constant conditions of pressure and temperature.
   b. The numerical value of this ratio is called the equilibrium constant for the given reaction.
3) The **equilibrium-constant expression** for a reaction is obtained by multiplying the concentrations of products, dividing by the concentrations of reactants, and raising each concentration to a power equal to its coefficient in the balanced chemical equation.
   a. Consider the following general equation: $aA + bB \rightleftharpoons yY + zZ$
4) The **equilibrium constant, $K_c$ (or $K_{eq}$)**, is the value obtained for the equilibrium–constant expression when equilibrium concentrations are substituted.
   a. The equilibrium constant for the general equation above is: $K_{eq} = \dfrac{[Y]^y[Z]^z}{[A]^a[B]^b}$
   b. A large $K_c$ (or $K_{eq}$) indicates large concentrations of products at equilibrium.

    c. A small $K_c$ (or $K_{eq}$) indicates large concentrations of unreacted reactants at equilibrium.

5) The **law of mass action** states that the value of the equilibrium constant expression, $K_c$, is constant for a particular reaction at a given temperature, <u>whatever</u> equilibrium concentrations are substituted.

## Introduction to the Reaction:

1) In this experiment, the equilibrium constant will be determined for the esterification reaction between acetic acid and isopropyl alcohol (2–propanol).



acetic acid    isopropyl alcohol        isopropyl acetate     water
$CH_3CO_2H$     (2–propanol)            i–PrAc       $H_2O$
**HAc**          **i–PrOH**

2) Using the abbreviations listed above, the equilibrium constant equation can be determined to be:

$$K_{eq} = \frac{[i-Pr\,Ac][H_2O]}{[HAc][i-Pr\,OH]}$$

3) This experiment will require two days/laboratory periods.
    a. During the first laboratory period, the initial concentration of acetic acid will be determined.
    b. After allowing time for the reaction mixture to reach equilibrium, the second laboratory period will focus on determining the equilibrium concentration of acetic acid.

4) The colorimetric titration process, which involves the delivery of a base to an acid to a colored end point with proper stoichiometric analysis, will be performed during both weeks to assess the acetic acid concentration.
    a. The titration reaction is as follows: $CH_3CO_2H(aq)$ + $NaOH(aq)$ → $CH_3CO_2Na(aq)$ + $H_2O(l)$

## Week 1 – Experimental Procedure for Initial Reaction Mixture:

1) Obtain one clean and completely dried 125 mL Erlenmeyer flask (with a rubber stopper), two 250 mL Erlenmeyer flasks, a buret with stand, two 100 mL beakers, and a 25 mL graduated cylinder.

2) Using a wax pencil, label the 125 mL Erlemeyer flask as "reaction mixture" and label the two 250 mL Erlemeyer flasks as "titration #1" and "titration #2".

3) Obtain approximately 100.0 mL of a standard 0.1000 *M* NaOH solution into one of the dry 100 mL beakers.

4) Place the empty 100 mL beaker under the buret.

5) Rinse the buret with approximately 10.0 mL of 0.1000 *M* NaOH solution and discard into the sink.
6) After rinsing, fill the buret with the remaining 0.1000 *M* NaOH solution to the 0.00 mL mark.
    a. Keep any remaining NaOH solution for later.
    b. Label beaker with "NaOH" with a wax pencil.
7) Place approximately 25.0 mL of deionized water into both titration Erlenmeyer flasks and set aside with the buret.
8) Using the 25 mL graduated cylinder (marked with 0.2 mL increments), measure 14.3 mL of glacial acetic acid and transfer into the 125 mL Erlemeyer flask.
    a. Record the volume with the highest accuracy.
9) Rinse and completely dry the graduated cylinder.
10) Using the 25 mL graduated cylinder (marked with 0.2 mL increments), measure 19.1 mL of isopropyl alcohol (2–propanol) and transfer into the 125 mL Erlemeyer flask.
    a. Record the volume with the highest accuracy.
11) Affix stopper to reaction flask and swirl for several minutes to mix reactants.
12) Using a 1.00 mL volumetric pipette with pipetting device or bulb, transfer 1.00 mL of the reaction mixture to each of the 250 mL Erlenmeyer flasks labeled "titration #1" and "titration #2".
13) Immediately restopper reaction flask to prevent evaporation.
14) Add 5–6 drops of phenolphthalein indicator to each titration flask.
    a. Note the initial color of both solutions in your lab notebook.
15) Record the initial level of the NaOH solution in the burette.
16) Place titration flask #1 under the tip of the burette and slowly begin to deliver the solution into the reaction mixture.
    a. Swirl the flask continually.
    b. You may add it quickly at first but slow the addition of NaOH when the pink color lingers.
    c. The endpoint of the titration is when the addition of a single added drop of NaOH solution produces a faint but permanent pink color.
17) Once the endpoint has been reached, record the final level of NaOH solution on the burette.
18) Refill the buret with NaOH to the 0.00 mL mark and repeat steps 15 – 17 with titration flask #2.
    a. Determine the average volume of NaOH used to titrate **1.00 mL** of reaction mixture.
19) Determine the molarity of acetic acid (HAc) in the reaction mixture.
    a. Moles of NaOH = [Molarity (moles/L)][Volume (L)]
    b. Moles of HAc = moles of NaOH used
    c. Molarity of HAc = (moles of HAc)/(volume of reaction mixture in Liters)
20) Discard titration flasks #1 & #2 into the sink.
21) Refill the burette with 0.1000 M NaOH solution.
22) Clean and dry the 1.00 mL volumetric pipette and both 250 mL Erlenmeyer flasks.
23) Add approximately 25.0 mL of deionized water to each titration flask.
24) Create an ice–water bath large enough to accommodate the 125 mL Erlenmeyer flask (reaction flask).

      a.   Add reaction flask to the ice–water bath and keep well stoppered.

      b.   This step will retard the rate of reaction as you add the sulfuric acid.

25) While swirling, add 5 drops of concentrated sulfuric acid to the reaction mixture.

      a.   Swirl well to ensure mixture becomes homogeneous.

26) Immediately after, pipette 1.00 mL of the reaction mixture to each of the 250 mL Erlenmeyer flasks labeled "titration #1" and "titration #2".

      a.   Note: These titrations should be done with some expedience because the reaction is proceeding more rapidly.

27) Re–stopper reaction mixture to prevent evaporation.

28) Repeat steps 14–18 above.

29) Calculate the volume of NaOH solution necessary to titrate the sulfuric acid by subtracting the mean volume used for acetic acid alone from the mean volume of the mixture of acids.

      a.   Note: The total volume of NaOH solution needed to titrate this new mixture should be LARGER than the mixture without the catalyst.

30) Do NOT discard the stoppered 125 mL Erlemeyer flask containing the "reaction mixture."

      a.   Store this mixture until next lab period as instructed by your professor.

31) Discard titration flasks #1 & #2 into the sink.


## Week 2 – Experimental Procedure for Equilibrium Reaction Mixture:

1) Obtain two clean and completely dried 250 mL Erlenmeyer flasks, a buret with stand, two 100 mL beakers, and a 25 mL graduated cylinder.

2) Using a wax pencil, label the two 250 mL Erlemeyer flasks as "titration #1" and "titration #2".

3) Obtain approximately 100.0 mL of a standard 0.1000 $M$ NaOH solution into one of the dry 100 mL beakers.

4) Place the empty 100 mL beaker under the buret.

5) Rinse the buret with approximately 10.0 mL of 0.1000 $M$ NaOH solution and discard into the sink.

6) After rinsing, fill the buret with the remaining 0.1000 $M$ NaOH solution to the 0.00 mL mark.

      a.   Keep any remaining NaOH solution for later.

      b.   Label beaker with "NaOH" with a wax pencil.

7) Place approximately 25.0 mL of deionized water into both titration Erlenmeyer flasks and set aside with the buret.

8) Using a 1.00 mL volumetric pipette with pipetting device or bulb, transfer 1.00 mL of the reaction equilibrium mixture to each of the 250 mL Erlenmeyer flasks labeled "titration #1" and "titration #2".

      a.   Using the waft test, note the odor of the reaction equilibrium mixture.

9) Immediately re–stopper reaction mixture to prevent evaporation.

10) Add 5–6 drops of phenolphthalein indicator to each titration flask.

      a.   Note the initial color of both solutions in your lab notebook.

11) Record the initial level of the NaOH solution in the burette.

12) Place titration flask #1 under the tip of the burette and slowly begin to deliver the solution into the reaction mixture.

    a. Swirl the flask continually.

    b. You may add it quickly at first but slow the addition of NaOH when the pink color lingers. BEWARE: The volume of NaOH needed will be LESS than in week 1.

    c. The endpoint of the titration is when the addition of a single added drop of NaOH solution produces a faint but permanent pink color.

13) Once the endpoint has been reached, record the final level of NaOH solution on the burette.

14) Refill the buret with NaOH to the 0.00 mL mark and and then repeat steps 12 – 13 with titration flask #2.

    a. Determine the average volume of NaOH used to titrate 1.00 mL of reaction equilibrium mixture.

15) Because sulfuric acid is a catalyst, it will have the same concentration (or volume required to neutralize) as last week.

16) Determine the molarity of acetic acid (HAc) in the equilibrium reaction mixture.

17) Discard titration flasks #1 & #2 into the sink.

18) Fill in the data table attached with the following information:

    a. Initial concentration of acetic acid, isopropyl alcohol, isopropyl acetate, and water.

    b. Equilibrium concentrations of acetic acid, isopropyl alcohol, isopropyl acetate, and water.

19) Calculate the equilibrium rate constant ($K_{eq}$) for this reaction at room temperature.

## Waste Disposal and Lab Clean – Up:

1) All equilibrium reaction materials should be placed into the appropriately labeled waste container in the hood.

2) All contents of titration flasks following neutralization with base can be disposed of via the sink.

3) Unused NaOH solution can be flushed down the sink.

## The Lab Write – Up:

1) Determine all values in your data table and discuss relevant data in your conclusion.

2) In your conclusion, provide the following values and discuss how the data was obtained:

    a. Initial concentration of acetic acid, isopropyl alcohol, isopropyl acetate, and water.

    b. Equilibrium concentrations of acetic acid, isopropyl alcohol, isopropyl acetate, and water.

3) Determine the value for $K_c$ (or $K_{eq}$) for this reaction at room temperature and discuss in your conclusion.

4) How would temperature have affected this equilibrium? Which direction would the equilibrium shift and why? How would the value for $K_c$ (or $K_{eq}$) change with temperature?

5) Using Le Chatelier's principle, discuss at least three ways to maximize the product yield. Explain each.

6) Discuss all sources of error during this experiment and how these errors directly affected the data.

# Sample Data Tables:

Initial Volumes/Concentration:
Volume of acetic acid (mL): _____

Volume of isopropyl alcohol (mL): _____

Concentration of standard NaOH solution (*M*): _____

| WEEK ONE (Acetic Acid Only) | Titration #1 | Titration #2 |
|---|---|---|
| Volume of reaction mixture titrated (mL) | 1.00 mL | 1.00 mL |
| Volume of NaOH solution used (mL) | | |
| Mean volume of NaOH solution (mL) | | |
| Moles of NaOH solution used (mol) | | |
| Moles of HAc present (mol) | | |
| Molarity of HAc (mol/L) | | |

| WEEK ONE (Acetic + Sulfuric) | Titration #1 | Titration #2 |
|---|---|---|
| Volume of reaction mixture titrated (mL) | 1.00 mL | 1.00 mL |
| Volume of NaOH solution used (mL) | | |
| Mean volume of NaOH solution (mL) | | |
| Volume correction to be applied in week 2 (mL) (Volume of NaOH necessary to react with sulfuric acid catalyst) | | |

Concentration of standard NaOH solution (*M*): _____

| WEEK TWO (Equilibrium) | Titration #1 | Titration #2 |
|---|---|---|
| Volume of reaction mixture titrated (mL) | 1.00 mL | 1.00 mL |
| Volume of NaOH solution used (mL) | | |
| **Corrected** volume of NaOH solution used to neutralize only HAc (mL) | | |
| **Corrected** mean volume of NaOH solution used to neutralize only HAc (mL) | | |
| Moles of HAc present at equilibrium (mol) | | |
| Molarity of HAc at equilibrium (mol/L) | | |

$$\text{HAc} + i\text{–PrOH} \rightleftharpoons i\text{–PrAc} + \text{H}_2\text{O}$$
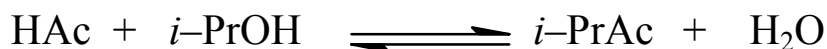
| | | | | | |
|---|---|---|---|---|---|
| **Initial** | | | | | |
| **Change** | | | | | |
| **Equilibrium** | | | | | |

| Compound Name | Initial Concentration (*M*) | Equilibrium Concentration (*M*) |
|---|---|---|
| **Acetic Acid** | | |
| **Isopropyl Alcohol** | | |
| **Isopropyl Acetate** | | |
| **Water** | | |

Equilibrium constant ($K_{eq}$): _____ @ __°C

# Experiment #5:
# Le Chatelier's Principle of Reaction Equilibrium for Complex–Ion Systems

## An Introduction to Le Chatelier's Principle:
1) All undisturbed reactions are at chemical equilibrium.
   a. Example: $CO(g) + 3\ H_2(g) \rightleftharpoons CH_4(g) + H_2O(g)$
2) Chemists attempt to maximize product yields by running the reaction under proper conditions.
   a. This will require a "disturbance" of the equilibrium to shift the reactions to favor the product side of the reaction.
   b. The following conditions can be changed to maximize product yields:
      i. Changing the concentrations of the product(s) by removal
      ii. Changing the concentrations of the reactant(s) by adding additional amounts
      iii. Changing the partial pressure of the gaseous reactants and products by modifying the volume
      iv. Changing the temperature of the reaction mixture
3) **Le Chatelier's principle** states that when a system at chemical equilibrium is disturbed by a change in temperature, pressure, or concentration, the equilibrium will shift to counteract this disturbance.

## The Conditions of Le Chatelier's Principle:
1) There are a number of conditions that can be changed that will disturb the equilibrium of a reaction.
2) Consider the equilibrium reaction depicted above: $CO(g) + 3\ H_2(g) \rightleftharpoons CH_4(g) + H_2O(g)$
3) **Reactant and product concentrations:**
   a. If the products (methane or water) were removed from the system at equilibrium, the reaction would shift to the right to produce more product.
   b. If additional amounts of reactants (carbon monoxide and/or hydrogen) were added, the reaction would again shift to the right to produce more product.
   c. Alternatively, the addition of additional amounts of product(s) or removal of reactant(s) would shift the equilibrium to the left.
4) **Volume effects:**
   a. A pressure change caused by changing the volume of the reaction vessel can affect the yield of products in a gaseous reaction only if the reaction involves a change in the total moles of gas present.
      i. In the equilibrium reaction above, four moles of gaseous reactants (1 mole of CO and 3 moles of $H_2$) are producing two moles of gaseous products (1 mole of $CH_4$ and 1 mole of $H_2O$).
         1. Thus, the products require less space than the reactants so reducing the volume of the reaction vessel would, therefore, favor the products.

2. The reverse holds if there are fewer moles of gaseous reactants than products.

5) **Pressure effects:**
   a. Increasing the pressure (or "squeezing") of the reaction mixture will cause a shift in the equilibrium toward the fewer moles of gas.
      i. Thus, in this equilibrium reaction above, increasing the pressure would shift the equilibrium left and increase concentrations of products.
   b. Conversely, reducing the pressure in the reaction vessel by increasing its volume would have the opposite effect.
   c. In the event that the number of moles of gaseous product equals the number of moles of gaseous reactant, vessel volume (and pressure) will have no effect on the position of the equilibrium.

6) **Temperature effects:**
   a. Temperature has a significant effect on most reactions.
      i. Reaction rates generally increase with an increase in temperature. Consequently, equilibrium is established sooner.
      ii. In addition, the numerical value of the equilibrium constant, $K_c$, varies with temperature.
   b. For an exothermic process:
      i. The change in enthalpy ($\Delta H$) is negative meaning that heat is released; heat, therefore, can be considered a product.
      ii. Increasing temperature would be analogous to adding more product, causing the equilibrium to shift left.
      iii. Since "heat" does not appear in the equilibrium-constant expression, this change would result in a smaller numerical value for $K_c$.
   c. For an endothermic process:
      i. The change in enthalpy ($\Delta H$) is positive meaning that heat is absorbed; heat, therefore, can be considered a reactant.
      ii. Increasing temperature would be analogous to adding more reactant, causing the equilibrium to shift right.
      iii. This change results in more product at equilibrium, and a larger numerical value for $K_c$.

## List of the Reactants:
1) 0.10 $M$ $Fe(NO_3)_3$
2) 0.10 $M$ KSCN
3) 0.10 $M$ $AgNO_3$
4) 0.10 $M$ $Cu(NO_3)_2$
5) 6.0 $M$ $NH_4OH$
6) 14.8 $M$ $HNO_3$ (concentrated)
7) 0.50 $M$ $Co(NO_3)_2$
8) 12.0 $M$ HCl (concentrated)

## Introduction to the Equilibrium Reactions:

### Part 1: The equilibrium between $Fe^{3+}$ and $SCN^-$ with $[FeSCN]^{2+}$:
1) The equilibrium between $Fe^{3+}(aq)$ and $SCN^-(aq)$ with $[FeSCN]^{2+}(aq)$ will be studied.
    a. The equilibrium will be assessed following the addition of extra $Fe^{3+}(aq)$ and $SCN^-(aq)$ compared to the reference.
    b. In addition, $Ag^+(aq)$ will be introduced to form the complex $[Ag(SCN)_4]^{3-}$
        i. An assessment to which thiocyanate complex (the iron or the silver) is more stable will be performed.
        ii. The most stable complex will have the largest value for the formation constant, $K_f$.

### Part 2: The equilibrium between $[Cu(H_2O)_4]^{2+}$ and $[Cu(NH_3)_4]^{2+}$:
1) A copper (II) complex with water and $NH_3$ will be studied and compared.
    a. The equilibrium between $[Cu(H_2O)_4]^{2+}(aq)$ and $[Cu(NH_3)_4]^{2+}(aq)$ will be established.
        i. Water and ammonia will be added to disturb the equilibrium.
        ii. In addition, nitric acid will be added and the observed shift of equilibrium will be assessed.

### Part 3: The equilibrium between $[Co(H_2O)_6]^{2+}$ and $[CoCl_4]^{2-}$:
1) The equilibrium between $[Co(H_2O)_6]^{2+}(aq)$ and $[CoCl_4]^{2-}(aq)$ will be studied.
    a. The equilibrium will be assessed at different temperatures to determine which complex is dominant.


## Experimental Procedure–Part 1
### The equilibrium between $Fe^{3+}$ and $SCN^-$ with $[FeSCN]^{2+}$:
1) Obtain 4 clean and completely dried test tubes, a $100 - 150$ mL beaker, a 10.0 mL graduated cylinder, a 25.0 mL graduated cylinder, and a glass stirring rod.
2) Using a wax/grease pencil, mark the numbers 1–4 on the test tubes for identification purposes.
3) Measure out 25.0 mL of deionized water in a graduated cylinder and transfer into the beaker.
4) Add 25 drops of 0.10 $M$ $Fe(NO_3)_3$ and 25 drops of 0.10 $M$ KSCN to the beaker.
5) Stir thoroughly with a stirring rod.
6) Add 4.0 mL of this solution into each of the test tubes using the 10.0 mL graduated cylinder.
7) To test tube #1, add 10 drops of deionized water and stir thoroughly. This is considered your reference point at which the equilibrium is undisturbed.
    a. Record color and clarity of solution.
8) To test tube #2, add 10 drops of 0.10 $M$ $Fe(NO_3)_3$ and mix thoroughly.
    a. Record color and clarity of solution.
9) To test tube #3, add 10 drops of 0.10 $M$ KSCN and mix thoroughly.

a. Record color and clarity of solution.
10) To test tube #4, add 10 drops of 0.10 $M$ $AgNO_3$ and mix thoroughly.
   a. Record color and clarity of solution.
11) Compare all four test tubes to one another by analyzing the intensity of the color.
   a. The larger the concentration of $Fe(SCN)^{2+}$, the more intense the original color.
   b. If results are unclear, place test tubes against a background of plain white paper.
12) To test tube #4, add 10 drops of 0.10 $M$ $Fe(NO_3)_3$ and mix thoroughly.
   a. Record color and clarity of solution.
   b. Note if the solution returns to the original color and make an assessment to which thiocyanate complex (the iron or the silver) is more stable.

## Experimental Procedure – Part 2
## The equilibrium between $[Cu(H_2O)_4]^{2+}$ and $[Cu(NH_3)_4]^{2+}$:
1) Obtain 8 clean and completely dried test tubes, a glass stirring rod, and a 10 mL graduated cylinder.
   a. Label the test tubes with a wax pencil as #1 – #8.
2) Note the color and clarity of the original copper (II) nitrate solution.
3) To all eight test tubes, add 20 drops of the 0.10 $M$ $Cu(NO_3)_2$.
4) To test tubes #1 – #4, add 20 drops of deionized water.
   a. Record color and clarity of solution.
   b. This is considered your reference point at which the equilibrium is undisturbed for $[Cu(H_2O)_4]^{2+}$.
5) To test tube #2, add 1.0 mL of deionized water.
   a. Record color and clarity of solution.
   b. Compare to reference.
6) To test tube #3, add 1.0 mL of the 0.10 $M$ $Cu(NO_3)_2$.
   a. Record color and clarity of solution.
   b. Compare to reference.
7) To test tube #4, add 1.0 mL of a 6.0 $M$ $NH_4OH$ (source of $NH_3$).
   a. Record color and clarity of solution.
   b. Compare to reference.
8) To test tubes #5 – #8, add 20 drops of a 6.0 $M$ $NH_4OH$ (source of $NH_3$).
   a. Record color and clarity of solution.
   b. This is considered your reference point at which the equilibrium is undisturbed for $[Cu(NH_3)_4]^{2+}$.
9) To test tube #6, add 1.0 mL of deionized water.
   a. Record color and clarity of solution.
   b. Compare to reference.
10) To test tube #7, add 1.0 mL of a 6.0 $M$ $NH_4OH$ (source of $NH_3$).
   a. Record color and clarity of solution.
   b. Compare to reference.
11) To test tube #8, add 1.0 mL of a 14.8 $M$ $HNO_3$.
   a. Record color and clarity of solution.
   b. Compare to reference.

# Procedure – Part 3
## The equilibrium between $[Co(H_2O)_6]^{2+}$ and $[CoCl_4]^{2-}$:

1) Obtain two clean and completely dried test tubes, a glass stirring rod, two 250 mL beakers, and a hot plate.
   a. Label test tubes as #1 and #2 with a wax pencil.
2) Create a hot water bath by filling the first 250 mL beaker to the 200 mL mark with water and placing onto the hot plate.*
   a. Heat until a moderate boil is achieved.
   b. *An alternate heating set–up can be implemented by using a ring stand and a Bunsen burner.
3) Create an ice water bath with the second 250 mL beaker by adding ice and water to the 200 mL mark.
4) Add 20 drops of 0.50 $M$ $Co(NO_3)_2$ to test tube #1.
   a. Record the color and clarity of the solution.
5) Add 20 drops of deionized water to test tube #1.
   a. Record the color and clarity of the solution.
   b. At room temperature, this is considered your reference point at which the equilibrium is undisturbed for the formation of $[Co(H_2O)_6]^{2+}$.
6) Add 20 drops of 0.50 $M$ $Co(NO_3)_2$ to test tube #2.
   a. Record the color and clarity of the solution.
7) Add 20 drops of concentrated 12.0 $M$ HCl to test tube #2 and swirl until homogeneous.
   a. Use glass stirring rod if necessary. Ensure that the glass rod is rinsed and dried after use due to the presence of concentrated acid.
   b. Record the color and clarity of the solution.
   c. Compare color & clarity to that of test tube #1.
8) To test tube #1, add 20 drops of deionized water and swirl until homogeneous.
   a. Use glass stirring rod if necessary. Ensure that the glass rod is rinsed and dried after use.
   b. Record the color and clarity of the solution.
9) To test tube #2, add 20 drops of deionized water and swirl until homogeneous.
   a. Use glass stirring rod if necessary. Ensure that the glass rod is rinsed and dried after use due to the presence of concentrated acid.
   b. At room temperature, consider this the equilibrium between $[CoCl_4]^{2-}$ and $[Co(H_2O)_6]^{2+}$. Compare color & clarity to that of test tube #1.
10) Using clamps, secure both test tubes to a ring stand and/or the metal scaffolding.
11) Place the test tubes into the boiling water for a few minutes until a color change has occurred.
    a. Record the color and clarity of each solution.
12) Allow test tubes to cool briefly but not to room temperature.
13) Next, immerse the test tubes into the ice water bath for a few minutes until a color change has occurred.
    a. Record the color and clarity of the solution.
14) Considering all data observed, determine how temperature affected the equilibrium and determine whether the formation of $[Co(H_2O)_6]^{2+}$ and $[CoCl_4]^{2-}$ are endothermic or exothermic.

**Waste Disposal and Lab Clean – Up**:
1) Place all heavy metal solutions into labeled waste containers.
2) Hot water and ice–water baths can be poured down the drain.

**The Lab Write – Up**:
1) Determine all values in your data table and discuss relevant data in your conclusion.
2) Provide equilibrium equations for each reaction under the balanced equation section.
3) For all parts, use Le Chatelier's principle to explain the different color and clarities and how they relate to the disturbance of the equilibrium in your conclusion.
   a. Discuss how the equilibrium was disturbed by each reaction process.
   b. For each reaction process, where the reactants or products favored?
4) For part 1, determine which thiocyanate complex (the iron or the silver) is more stable. Which has a larger $K_f$? Explain.
5) For part 2, which complex has a larger $K_f$, $[Cu(H_2O)_4]^{2+}(aq)$ or $[Cu(NH_3)_4]^{2+}(aq)$? How did $HNO_3$ affect the equilibrium for $[Cu(NH_3)_4]^{2+}(aq)$? Provide a reaction to explain.
6) For part 3, is the formation of $[Co(H_2O)_6]^{2+}(aq)$ endothermic or exothermic? Is the formation of $[CoCl_4]^{2-}(aq)$ endothermic or exothermic? Explain by how temperature modified the color.
7) Discuss all sources of error during this experiment and how they directly affected the data.

# The Balanced Equilibrium Equations:

| Part | Reactant(s) | | Product(s) |
|---|---|---|---|
| 1 | ___ $Fe^{3+}(aq) +$ ___ $SCN^-(aq)$ | ⇌ | |
| 1 | ___ $Ag^+(aq) +$ ___ $SCN^-(aq)$ | ⇌ | |
| 1 | ___ $[FeSCN]^{2+}(aq) +$ ___ $Ag^+(aq)$ | ⇌ | |
| 1 | ___ $[Ag(SCN)_4]^{3-}(aq) +$ ___ $Fe^{3+}(aq)$ | ⇌ | |
| | | | |
| 2 | ___ $Cu^{2+}(aq) +$ ___ $H_2O(l)$ | ⇌ | |
| 2 | ___ $Cu^{2+}(aq) +$ ___ $NH_3(aq)$ | ⇌ | |
| 2 | ___ $[Cu(H_2O)_4]^{2+}(aq) +$ ___ $NH_3(aq)$ | ⇌ | |
| 2 | ___ $[Cu(NH_3)_4]^{2+}(aq) +$ ___ $H_2O(l)$ | ⇌ | |
| 2 | ___ $HNO_3(aq) +$ ___ $NH_3(aq)$ | → | |
| 2* | ___ $NH_4OH(aq)$ | ⇌ | |
| | | | |
| 3 | ___ $Co^{2+}(aq) +$ ___ $H_2O(l)$ | ⇌ | |
| 3 | ___ $Co^{2+}(aq) +$ ___ $Cl^-(aq)$ | ⇌ | |
| 3 | ___ $[Co(H_2O)_6]^{2+}(aq) +$ ___ $Cl^-(aq)$ | ⇌ | |
| 3 | ___ $[CoCl_4]^{2-}(aq) +$ ___ $H_2O(l)$ | ⇌ | |

**\*Side reaction**

# Sample Data Tables:

**Part 1 – The equilibrium between $Fe^{3+}$ and $SCN^-$ with $[FeSCN]^{2+}$:**

| Test Tube No. | Reagent Information | Color & Clarity |
|---|---|---|
| 1 | Reference/10 drops $H_2O$ | |
| 2 | 10 drops 0.10 $M$ $Fe(NO_3)_3$ | |
| 3 | 10 drops 0.10 $M$ KSCN | |
| 4a | 10 drops 0.10 $M$ $AgNO_3$ | |
| 4b | 4a + 10 drops 0.10 $M$ $Fe(NO_3)_3$ | |

Comparison between:

1 & 2: _____    1 & 3: _____

1 & 4a: _____    1 & 4b: _____

**Part 2 – The equilibrium between $[Cu(H_2O)_4]^{2+}$ and $[Cu(NH_3)_4]^{2+}$:**

**Color & Clarity of Original $Cu(NO_3)_2$ Solution: _____**

| Test Tube No. | Reagent Information | Color & Clarity |
|---|---|---|
| 1 | Reference (A) for $[Cu(H_2O)_4]^{2+}$: 20 drops of 0.10 $M$ $Cu(NO_3)_2$ + 20 drops of deionized water | |
| 2 | Reference (A) + 1.0 mL of deionized water | |
| 3 | Reference (A) + 1.0 mL of 0.10 $M$ $Cu(NO_3)_2$ | |
| 4 | Reference (A) + 1.0 mL of 6.0 $M$ $NH_4OH$ | |
| 5 | Reference (B) for $[Cu(NH_3)_4]^{2+}$: 20 drops of 0.10 $M$ $Cu(NO_3)_2$ + 20 drops of 6.0 $M$ $NH_4OH$ | |
| 6 | Reference (B) + 1.0 mL of deionized water | |
| 7 | Reference (B) + 1.0 mL of 6.0 $M$ $NH_4OH$ | |
| 8 | Reference (B) + 1.0 mL of 14.8 $M$ $HNO_3$ | |

# Part 3 – The equilibrium between $[Co(H_2O)_6]^{2+}$ and $[CoCl_4]^{2-}$:

### Test Tube #1:

| Data Point No. | Reagent Information/Temp. | Color & Clarity |
|---|---|---|
| 1 | 20 drops of 0.50 $M$ Co(NO$_3$)$_2$ | |
| 2 | $[Co(H_2O)_6]^{2+}$ reference: <br> + 20 drops of deionized water | |
| 3 | + 20 drops of deionized water | |
| 4 | Hot (~100$^o$C) | |
| 5 | Cold (~0$^o$C) | |

### Test Tube #2:

| Data Point No. | Reagent Information/Temp. | Color & Clarity |
|---|---|---|
| 1 | 20 drops of 0.50 $M$ Co(NO$_3$)$_2$ | |
| 2 | $[CoCl_4]^{2-}$ reference: <br> + 20 drops of 12.0 $M$ HCl | |
| 3 | + 20 drops of deionized water | |
| 4 | Hot (~100$^o$C) | |
| 5 | Cold (~0$^o$C) | |

# Experiment #6:
# The Relative Strengths of Acids;
# An Introduction to Acidity and pH

**An Introduction to the Acid–Base Theories**:
1) There are three theories that classify acids and bases.
   a. The **Arrhenius** concept
      i. In this theory, an acid is a substance that, when dissolved in water, increases the concentration of hydronium ion ($H_3O^+$).
      ii. A base, in the Arrhenius concept, is a substance that, when dissolved in water, increases the concentration of hydroxide ion, $OH^-(aq)$.
   b. The **Brønstead–Lowry** concept
      i. An **acid** is the species donating a proton in a proton–transfer reaction.
      ii. A **base** is the species accepting a proton in a proton–transfer reaction.
   c. The **Lewis** concept
      i. In the Lewis concept, an acid is defined as an electron pair acceptor and a base is an electron pair donor.
2) The Brønsted–Lowry concept introduces the idea of **conjugate acid–base pairs** in proton–transfer reactions.
3) A conjugate acid – base pair consists of two species in an acid–base reaction, the first an acid and the second a base, that differ by the loss or gain of a proton.
4) When the acid–base reaction is reversible, both forward and backward directions involve proton transfer reactions.
   a. Example: $NH_3(aq) + H_2O(l) \rightleftharpoons NH_4^+(aq) + {}^-OH(aq)$
      i. In the forward direction, the ammonia ($NH_3$) functions as a base and water ($H_2O$) as the acid.
      ii. In the forward direction, the ammonium ion ($NH_4^+$) functions as a conjugate acid and hydroxide ion ($^-OH$) as the conjugate base.
      iii. $NH_4^+$ and $NH_3$ is an example of a conjugate acid–base pair.

**The Self – Ionization of Water and an Introduction to pH:**
1) Although it has a small equilibrium constant, water autoionizes into the hydroxide and hydronium ions as shown by the following equilibrium reaction:
   a. $H_2O(l) + H_2O(l) \rightleftharpoons H_3O^+(aq) + OH^-(aq)$
2) With this equation, the equilibrium express for the self–ionization is as follows:
   a. $$K_c = \frac{[H_3O^+][OH^-]}{[H_2O]^2}$$
3) The concentration of ions is extremely small, so the concentration of $H_2O$ remains essentially constant; thus, the equilibrium expression can be written as:
   a. $[H_2O]^2 K_c = [H_3O^+][OH^-]$
   b. $[H_2O]^2 K_c$ is a constant and is called the **ion–product constant for water,** which is depicted as $K_w$.
   c. $K_w = [H_3O^+][OH^-]$
   d. At 25 $^o$C, the value of $K_w$ is $1.0 \times 10^{-14}$.

i.  Like any equilibrium constant, $K_w$ varies with temperature.
4) Because of this self–ionization, all aqueous solutions, whether they are acidic, neutral, or basic, can be compared by expressing their hydronium ion concentration.
    a.  For acidic solutions: $[H_3O^+] > 1.0 \times 10^{-7}\ M$
    b.  For neutral solutions: $[H_3O^+] = 1.0 \times 10^{-7}\ M$
    c.  For basic solutions: $[H_3O^+] < 1.0 \times 10^{-7}\ M$
5) These concentrations are small so it is more convenient to express acidity in terms of pH.
    a.  pH is defined as the negative logarithm of the molar hydronium ion concentration.
    b.  $pH = -\log[H_3O^+]$

## The Relative Strength of Acids:
1) In the Brønsted–Lowry concept, it is practical to consider acid–base reactions as a competition for protons.
2) Acids can be ranked by their relative strengths as proton donors.
3) Stronger acids will lose protons more effectively than weaker ones.
    a.  Thus, strong acids completely ionize when dissolved in water and the reverse reaction only occurs to a small extent.
    b.  Example: $HClO_4(aq) + H_2O(l) \rightarrow ClO_4^-(aq) + H_3O^+(aq)$
4) There are two factors that govern the strength of an acid; bond polarity and bond strength.
    a.  The more polarized the bond, the more the electrons within the sigma bond are unequally distributed, and the easier the hydrogen can be removed.
        i.  Bond polarity is directly related to the dipole created from differences in atomic electronegativities.  The larger the electronegativity difference, the stronger the acid.
    b.  The weaker the bond, the easier the proton is removed, and the stronger the acid.
        i.  Bond strength is inversely proportional to the size of the atom bonded to the hydrogen.
        ii.  The larger the atom bonded to the hydrogen, the weaker the bond, and the stronger the acid.
5) For a series of oxoacids, the acid strengths increase as the number of oxygen atoms bonded to the central atom attached to the hydrogen.
    a.  Because the central atom becomes more electronegative with increasing oxygens, the oxoacid becomes stronger.

## The Acids to Assess:
1) $B(OH)_3$ (0.10 $M$ and 0.50 $M$)
2) $HNO_3$ (0.10 $M$ and 0.50 $M$)
3) $H_2SO_4$ (0.10 $M$ and 0.50 $M$)
4) $H_3PO_4$ (0.10 $M$ and 0.50 $M$)
5) $HC_2H_3O_2$ (0.10 $M$ and 0.50 $M$)
6) $H_2O$
7) Fruit Juices (Apple, Grapefruit, Orange, Tomato)
8) Vinegar
9) Ultra Bleach
10)  Diet Pepsi®

**Pre–Lab Information**:
1) Obtain the values for electronegativity for hydrogen, oxygen, nitrogen, phosphorus, carbon, sulfur, and boron.  Record in data table and provide an appropriate reference.
2) Determine the IUPAC names for the first five acids listed and provide the Lewis–Dot structures for each.  Record in data table.
3) Note: Proper lab attire is required for this experiment.  Students not dressed appropriately will not be permitted into the lab.

**Procedure for Assessing pH**:
1) Obtain 17 clean and completely dried test tubes, one clean and dried 10.0 mL graduated cylinder, and one glass stirring rod.
   a. It would be advisable to line your workspace with paper towels.
2) Obtain wide range pH paper and a pH probe (or pH meter).
   a. If advised, calibrate the pH probe with the buffer solutions provided.
3) Remove the pH probe from the electrode storage solution and rinse well with deionized/distilled water to achieve a pH of around 7.0.
   a. Do not discard electrode solution unless advised.
4) Soak the pH probes in deionized/distilled water in a 250 mL beaker.
   a. Record the pH value in the data table.
5) With a wax pencil, label each of the test tubes by their molarity and acidic identity.
6) Place approximately 5–10 mL of each acid into their labeled test tube.
7) Obtain a paper towel on which to place a piece of pH paper.
8) Insert the glass stirring rod into the first solution and transfer a drop of the acid onto the pH paper.
9) With the color guide attached, record the pH with the greatest accuracy.
   a. Place this value in the data table.
10) Next, carefully dry off the probe with a Kimwipe, insert the pH probe into the solution, and allow the for a stable pH value.
    a. Record this value in the data table.
11) Using deionized/distilled water, thoroughly rinse off the pH probe and then store in a 250 mL beaker of deionized water with the probe completely immersed.
12) Repeat steps 8–11 with all remaining solutions, including the household solutions.
    a. Do not forget to rinse off probe and dry with a Kimwipe between each measurement.
13) For all of the acids (and molarities), calculate the hydronium ion concentrations based on the probe measurements.

**Waste Disposal**:
1) All acids should be placed into the appropriately labeled waste container for neutralization and disposal.
2) Return pH probe to the electrode storage solution.
3) All household products can be placed down the sink.

**The Lab Write – Up**:

1) Determine all values in your data table and discuss relevant data in your conclusion.
2) Discuss the precision and accuracy of using pH paper versus the pH probes providing empirical evidence.
3) For each set of molarities (0.10 $M$ and 0.50 $M$), place the following acidic solutions in order of increasing acidity: $B(OH)_3$, $HNO_3$, $H_2SO_4$, $H_3PO_4$, $HC_2H_3O_2$.
   a. Place them in the correct order if above is incorrect.
   b. Discussing molecular structure, bond polarity, and bond strength, provide reasons how and why this order of acidity can be predicted.
4) Place the fruit juices in order of increasing acidity. Which fruit juice would be most troublesome for patients suffering from acid–reflux disease?
5) How does the acidity of household vinegar compare to the 0.10 $M$ and 0.50 $M$ solutions? Speculate on an approximate molarity.
6) The active ingredient in household bleach is sodium hypochlorite. Show the balanced equilibrium reaction by which this produces hypochlorous acid. In this concentration, how does it compare to the acids in question #3?
7) There are three main acidic compounds in Diet Pepsi®. What are these compounds? Show the balanced equation by which the one gaseous acidic oxide produces an acid when dissolved in water. Based on the pH and other factors, what health consequences are in store for those that drink this beverage regularly?
8) Discuss all sources of error during this experiment and how the error directly affects the results.

# Pre–Lab Sample Data Tables:

| Element | Electronegativity |
|---------|-------------------|
| H       |                   |
| B       |                   |
| C       |                   |
| N       |                   |
| O       |                   |
| S       |                   |
| P       |                   |

Reference(s): _____

_____

| Molecular Formula | IUPAC Name | Lewis–Dot Structure |
|-------------------|------------|---------------------|
| $B(OH)_3$         |            |                     |
| $HNO_3$           |            |                     |
| $H_2SO_4$         |            |                     |
| $H_3PO_4$         |            |                     |
| $HC_2H_3O_2$      |            |                     |

# Sample Data Tables:

| Molecular Formula | Molarity | pH via Paper | pH via Probe | $[H_3O^+]$ |
|---|---|---|---|---|
| $H_2O$ | N/A | N/A | | |
| $B(OH)_3$ | 0.10 $M$ | | | |
| $HNO_3$ | 0.10 $M$ | | | |
| $H_2SO_4$ | 0.10 $M$ | | | |
| $H_3PO_4$ | 0.10 $M$ | | | |
| $HC_2H_3O_2$ | 0.10 $M$ | | | |

| Molecular Formula | Molarity | pH via Paper | pH via Probe | $[H_3O^+]$ |
|---|---|---|---|---|
| $B(OH)_3$ | 0.50 $M$ | | | |
| $HNO_3$ | 0.50 $M$ | | | |
| $H_2SO_4$ | 0.50 $M$ | | | |
| $H_3PO_4$ | 0.50 $M$ | | | |
| $HC_2H_3O_2$ | 0.50 $M$ | | | |

# Sample Data Tables Continued:

| Juice | pH via Paper | pH via Probe | $[H_3O^+]$ |
|---|---|---|---|
| Apple | | | |
| Grapefruit | | | |
| Orange | | | |
| Tomato | | | |

Increasing order of fruit juice acidity from least acidic to the most acidic:

_____ < _____ < _____ < _____

| Household Material | pH via Paper | pH via Probe | $[H_3O^+]$ |
|---|---|---|---|
| Vinegar | | | |
| Bleach | | | |
| Diet Pepsi® | | | |

# Experiment #7:
# Determination of the Molar Mass and $K_a$ for a Weak Monoprotic Acid

**An Introduction to Acid–Base Equilibrium**:

1) An acid reacts with water to produce hydronium ions (hydrogen ions) and a conjugate base ion.
    a. This process is called **acid ionization** or acid dissociation.
    b. An example acid ionization equilibrium for acetic acid is depicted below:
        i. $HC_2H_3O_2(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + C_2H_3O_2^-(aq)$
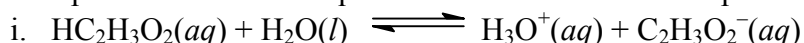
2) For strong acids (strong electrolytes), the ion concentrations are determined by the stoichiometry of the reaction from the original concentration.

3) For weak acids (weak electrolytes), the ion concentrations are determined by the acid–ionization constant (or acid–dissociation constant).

4) The equilibrium constant expression for the acetic acid equilibrium shown above is as follows:

$$K_c = \frac{[H_3O^+][C_2H_3O_2^-]}{[HC_2H_3O_2][H_2O]}$$

5) If dilute solution of this weak acid is used, the concentration of water is nearly constant and thus can be factored into the equilibrium constant ($K_c$).

6) The $K_a$ value is the acid–ionization constant which assumes that the concentration of water is constant:

$$Ka = \frac{[H_3O^+][C_2H_3O_2^-]}{[HC_2H_3O_2]}$$

7) An experimental determination of $K_a$ can be performed using two methods:
    a. Method #1: Electrical conductivity or measure of a colligative property to determine degree of ionization.
        i. The **degree of ionization** of a weak electrolyte is the fraction of molecules that react with water to produce ions.
            1. This may be expressed as a percent ionization.
    b. Method #2: pH determination
        i. In this method, the pH is determined to calculate $[H_3O^+]$ and consequently the concentrations of other ions.

**The Self – Ionization of Water and an Introduction to pH:**

1) Although it has a small equilibrium constant, water autoionizes into the hydroxide and hydronium ions as shown by the following equilibrium reaction:
    a. $H_2O(l) + H_2O(l) \rightleftharpoons H_3O^+(aq) + OH^-(aq)$

2) With this equation, the equilibrium express for the self–ionization is as follows:
    a. $K_c = \frac{[H_3O^+][OH^-]}{[H_2O]^2}$

3) The concentration of ions is extremely small, so the concentration of $H_2O$ remains essentially constant; thus, the equilibrium expression can be written as:

a. $[H_2O]^2 K_c = [H_3O^+][OH^-]$
b. $[H_2O]^2 K_c$ is a constant and is called the **ion–product constant for water,** which is depicted as $K_w$.
c. $K_w = [H_3O^+][OH^-]$
d. At 25 °C, the value of $K_w$ is 1.0 x $10^{-14}$.
    i. Like any equilibrium constant, $K_w$ varies with temperature.
4) Because of this self–ionization, all aqueous solutions, whether they are acidic, neutral, or basic, can be compared by expressing their hydronium ion concentration.
    a. For acidic solutions: $[H_3O^+] > 1.0$ x $10^{-7} M$
    b. For neutral solutions: $[H_3O^+] = 1.0$ x $10^{-7} M$
    c. For basic solutions: $[H_3O^+] < 1.0$ x $10^{-7} M$
5) These concentrations are small so it is more convenient to express acidity in terms of pH.
    c. pH is defined as the negative logarithm of the molar hydronium ion concentration.
    d. $pH = -\log[H_3O^+]$


## The Acid–Ionization of a Weak Monoprotic Acid (HA):
1) The generic equation and the acid–ionization constant expression is depicted below.
    a. $HA(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + A^-(aq)$ $\qquad$ $K_a$

    b. $$K_a = \frac{[H_3O^+][A^-]}{[HA]}$$


## The Acid – Base Neutralization Reaction via Titration:
1) The generic equation for the acid–base neutralization reaction performed via titration is depicted below.
    a. $HA(aq) + NaOH(aq) \rightleftharpoons H_2O(l) + NaA(aq)$ $\qquad$ $K_a$


## The Unknown Weak Monoprotic Acids:
1) Acetylsalicylic acid (aspirin)
    a. Acetylsalicylic acid (aspirin) is a common over–the–counter (OTC) drug used as analgesic, antipyretic, antiplatelet agent, and anti–inflammatory drug.
2) Nicotinic acid (niacin)
    a. Nicotinic acid (niacin) is a water–soluble B–complex vitamin that is an essential human nutrient obtained via the diet.
    b. Under a physician's care, it can be used to treat high cholesterol.

**Data Interpretation to Determine the Molar Mass & $K_a$:**

1) Following the titration of the weak monoprotic acid solution with standardized sodium hydroxide (NaOH) solution, two plots will be constructed.
   a. Graph #1: A plot of the volume of NaOH added versus the solution pH.
   b. Graph #2: A plot of the volume of NaOH added versus the change in pH.
      i. This is the change from the *previous pH value.*
2) To determine the molar mass of the unknown monoprotic acid, the equivalence point must be determined.
   a. The **equivalence point** is the point in a titration in which an exact stoichiometric amount of a reagent is added to neutralize.
   b. Using Graph #2, the equivalence point on the graph is the exact volume of NaOH which produce maximum pH changes.
      i. This is often referred to as the inflection point which, in calculus, denotes the point on a curve in which the concavity changes sign.
   c. Once the equivalence point volume has been determined, this volume is used to determine the equivalence point pH using Graph #1.
3) To determine the $K_a$ using the titration plots, the equivalence point must be determined as discussed above.
   a. At half of the equivalence point volume added, $[HA] = [A^-]$.
   b. Using the acid equilibrium expression, at this halfway point, the $K_a = [H_3O^+]$
      i. Remember that $pH = -\log[H_3O^+]$ so $[H_3O^+] = \text{antilog}(-pH)$
   c. At this halfway point, the $pK_a = -\log K_a$.

## Graph #1: Plot of the Volume of NaOH Added Versus the Solution pH
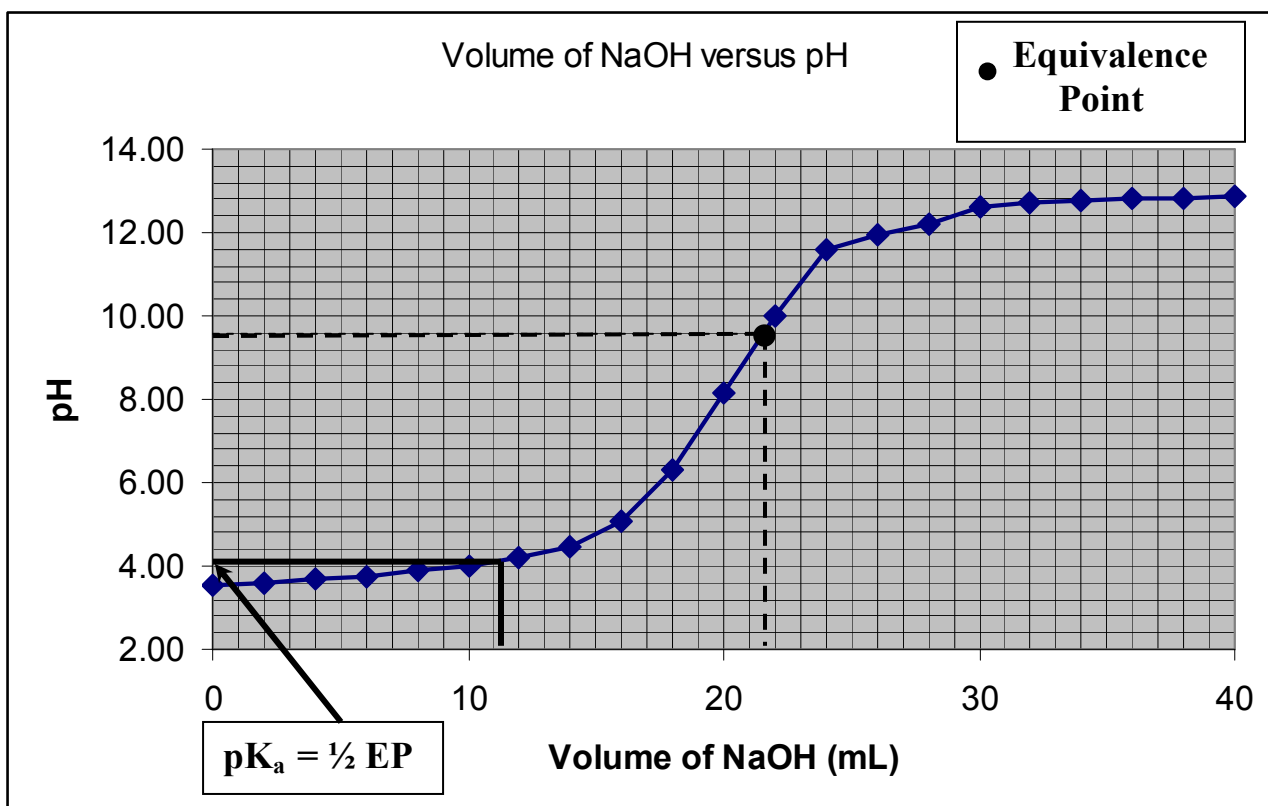
## Graph #2: Plot of the Volume of NaOH Added Versus the Change in pH
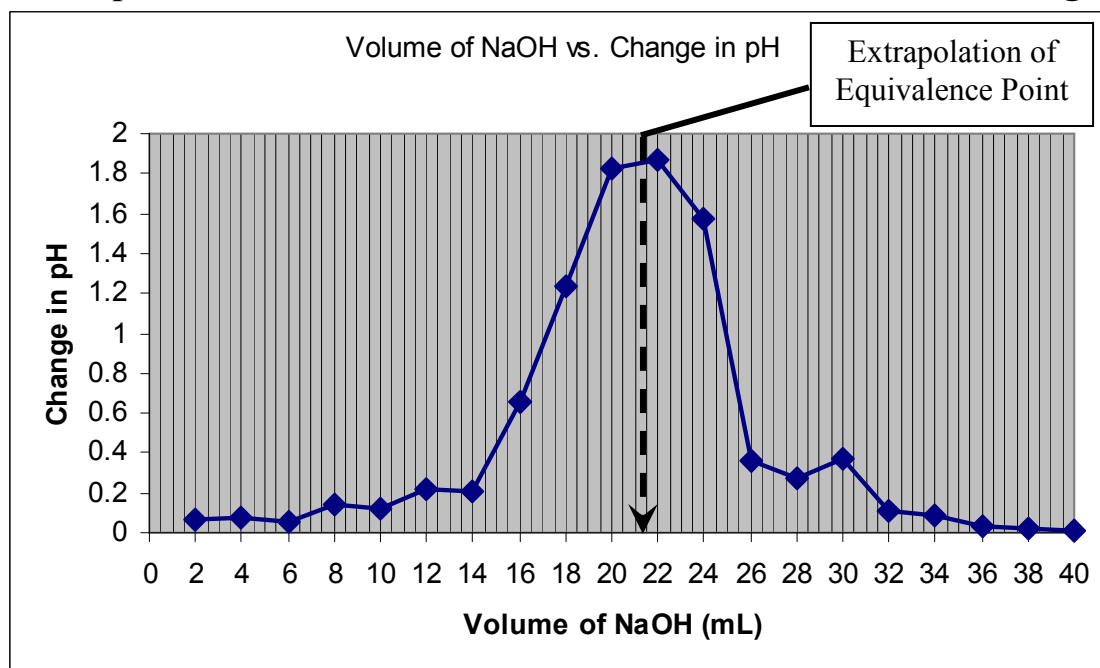


**Pre–Lab Information**:
1) Obtain the true values for the $K_a$ and p$Ka$ for acetylsalicylic acid (aspirin) and nicotinic acid (niacin).
   a. Provide a reference for these values.
   b. These true values will be necessary to compare with experimental results.


**Experimental Procedure**:
1) Obtain two clean and completely dry 250 mL beakers, a 100 mL beaker, a buret with buret clamp, a ring stand, a hot plate with a stir function, a magnetic stir bar, one clean and dried 100.0 mL graduated cylinder, a short-stem funnel, a utility clamp, and a glass stirring rod.
2) Obtain a pH probe (or pH meter).
   a. If advised, calibrate the pH probe with the buffer solutions provided.
3) Remove the pH probe from the electrode storage solution and rinse well with deionized/distilled water to achieve a pH of around 7.0.
   a. Do not discard electrode solution unless advised.
4) Soak the pH probes in deionized/distilled water in a 250 mL beaker.
   a. Record the pH value in the data table.
5) Add 100.0 mL of deionized/distilled water to the 100.0 mL graduated cylinder and transfer into a clean and dry 250 mL beaker.
6) Place beaker onto a stirring hotplate. Add magnetic stir bar and set to a gentle stir.
   a. You will NOT need the heating function for the hot plate.

7) Measure *no more* than 0.300 g of your unknown monoprotic acid on weighing paper using an analytical balance.
   a. Record the exact mass and unknown letter/number in your lab notebook.
   b. It is advised to weigh out somewhere between 0.250 – 0.280 g of your unknown monoprotic acid to ensure solubility.
8) Place the unknown monoprotic acid into the 250 mL beaker and allow to completely dissolve.
9) Obtain approximately 60.0 mL of a standard 0.1000 *M* NaOH solution into a dry 100 mL beaker.
   a. Label beaker as "NaOH."
   b. Record this standardized concentration in your data table.
10) Ensure the stopcock is in the closed position. Using a short stem funnel, add approximately 10.0 mL of the standardized NaOH solution to the buret.
11) Condition the buret by holding it horizontally and rotating to allow the NaOH solution to wet the inside of the glass completely. Discard the rinse into the sink.
12) Fill the buret with standardized NaOH solution and adjust the meniscus to zero.
   a. Record the initial buret reading as 0.00 mL.
13) Carefully dry off pH probe with a Kimwipe.
14) Next, using a utility clamp, suspend the pH probe to one side of the beaker into the acid solution and allow the for a stable pH value.
   b. Record this value in the data table.
   c. DO NOT allow the pH probe to come into contact with the magnetic stir bar.
15) Suspend the buret over the acid solution to the side opposite the pH probe.
16) Add approximately 1.00 mL of the NaOH solution and allow the pH to stabilize.
   a. Upon delivering the sodium hydroxide solution to the acid, it is critical that the pH probe test the solution and not the dripping base.
   b. Record the new buret reading and the pH in your data table.
   c. In addition, calculate change in pH from the previous reading.
17) Repeat step 16 until the change in pH is greater than 0.25 or $\Delta pH > 0.25$.
18) When the $\Delta pH > 0.25$, begin adding only 0.25–0.5 mL allotments and allow the pH to stabilize.
   a. Record the new buret reading and the pH in your data table.
   b. In addition, calculate change in pH from the previous reading.
19) Repeat step 18 until the change in pH is less than 0.25 or $\Delta pH < 0.25$.
20) When the $\Delta pH < 0.25$, begin adding 1.0 mL allotments and allow the pH to stabilize.
   a. Record the new buret reading and the pH in your data table.
   b. In addition, calculate change in pH from the previous reading.
21) Once ~30.0 mL of NaOH solution has been added and the pH is severely basic, the titration is complete.
22) Repeat for a second trial.
23) Remove the pH probe and rinse thoroughly with deionized/distilled water. Dry with a Kimwipe and then return to the electrode storage solution.
24) Remove the NaOH solution from the buret and carefully clean.
25) Construct a plot of the volume of NaOH added versus the solution pH.
   a. Place the volume of NaOH on the x–axis and pH on the y–axis.
26) Create a graph of the change in pH ($\Delta pH$) versus volume of NaOH added.

a. Place the volume of NaOH on the x–axis and $\Delta$pH on the y–axis.
b. Focus on plotting the area in which the change of pH is steep.
c. Extrapolate the equivalence point by determining the maxima.


## Waste Disposal and Lab Clean–Up:
1) All acidic solutions that have been effectively neutralized with sodium hydroxide solution can be placed down the drain.
2) Unused NaOH solution can be flushed down the sink with plenty of water.


## The Lab Write – Up:
1) Determine all values in your data table and discuss relevant data in your conclusion.
2) Construct a plot of the volume of NaOH added versus the solution pH.
3) Create a graph of the change in pH ($\Delta$pH) versus volume of NaOH added.
4) Determine the molar mass and identity of the weak monoprotic acid. Calculate the percent error and discuss reasons for error.
   a. Percent error = [(true value – experimental value)/true value] * 100%
   b. Percent error cannot be a negative value. Take the absolute value of the number if necessary.
5) Determine the $K_a$ of the weak monoprotic acid and compare to the true value.
6) Calculate the percent error, discuss all reasons for error, and include how these errors affected the data.
7) What assumptions were made during this experiment?
8) Could this same procedure (to determine the $K_a$ and molar mass) have been performed using sulfuric acid ($H_2SO_4$) as the acid? Explain why or why not.


# Pre–Lab Information:

| Drug Name | Molar Mass (g/mol) | $K_a$ | $pK_a$ |
|---|---|---|---|
| Acetylsalicylic acid (Aspirin) | | | |
| Nicotinic acid (Niacin) | | | |

References: _____

_____

# Sample Data Tables:

## Trial 1

| pH | ΔpH | Volume of NaOH Added (mL) |
|---|---|---|
| | 0 | 0.00 |
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# Sample Data Tables Continued:

## Trial 2

| pH | ΔpH | Volume of NaOH Added (mL) |
|---|---|---|
|  | 0 | 0.00 |
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# Sample Data Tables Continued:

| Data Recorded/Calculated | Trial #1 | Trial #2 |
|---|---|---|
| a) Unknown monoprotic acid number/letter: | | |
| b) Mass of unknown monoprotic acid in grams: | | |
| | | |
| c) Initial pH of water soaking solution: | | |
| d) Initial pH of unknown monoprotic acid solution: | | |
| | | |
| e) Standardized molar concentration of NaOH used in moles per liter: | | |
| | | |
| f) Equivalence point volume of NaOH (determined by graph) in milliliters: | | |
| g) Moles of NaOH at the equivalence point in mol*: | | |
| | | |
| h) Moles of unknown monoprotic acid titrated in mol[†]: | | |
| i) Molar mass of unknown monoprotic acid (b/h): | | |
| j) Average molar mass of unknown monoprotic acid in grams per mole ([trial 1 + 2]/2): | | |
| k) Identity of unknown monoprotic acid: | | |
| l) True molar mass of unknown monoprotic acid in grams per mole: | | |
| m) Percent error for molar mass in %: | | |
| | | |
| n) Equivalence point volume of NaOH (determined by graph) in milliliters (see f): | | |
| o) Volume of NaOH at half the equivalence point in milliliters (n/2): | | |
| p) Experimental p$K_a$ of unknown monoprotic acid[††]: | | |
| q) Average experimental p$K_a$ of unknown monoprotic acid ([trial 1 + 2]/2): | | |
| r) True p$K_a$ of unknown monoprotic acid: | | |
| s) Percent error for the p$K_a$ in %: | | |
| | | |
| t) Experimental $K_a$ of unknown monoprotic acid[†††]: | | |
| u) Average $K_a$ of unknown monoprotic acid ([trial 1 + 2]/2): | | |
| v) True $K_a$ of unknown monoprotic acid: | | |
| w) Percent error for the $K_a$ in %: | | |

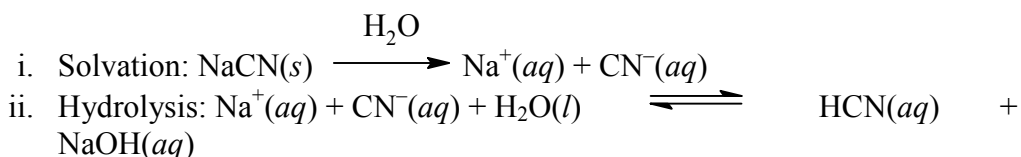| | |
|---|---|
| *Using standardized molarity of NaOH in data point e)<br>[†]Using the mole to mole ratio of NaOH to HA | [††]Using the pH at half of the equivalence point volume of NaOH<br>[†††]$Ka$ = antilog($-pKa$) |

# Experiment #8:
# Determination of Unknown Aqueous Salt Solutions by pH and Analysis of Buffers

**Introduction to Acid–Base Properties of Salt Solutions**:
1) Salts may be considered as products from an acid – base neutralization.
2) Often, these salts are acidic or basic when placed into an aqueous media due to ion hydrolysis.
3) **Ion hydrolysis** is the reaction in which an ion with water produces the conjugate acid and hydroxide ion or the conjugate base and hydronium ion.
   a. Example: Sodium cyanide (NaCN)
   b. A 0.10 M solution of NaCN has a pH of 11.1.
   c. The processes of solvation and hydrolysis are shown below:

   i. Solvation: $NaCN(s) \xrightarrow{\text{H}_2\text{O}} Na^+(aq) + CN^-(aq)$
   ii. Hydrolysis: $Na^+(aq) + CN^-(aq) + H_2O(l) \rightleftharpoons HCN(aq) + NaOH(aq)$
   d. This salt can be considered one from the neutralization of a strong base (NaOH) with a weak acid (HCN).
4) The acidity, basicity, or neutrality of a salt can be predicted from the relative strengths of the acids and bases that underwent neutralization to produce that salt.
   a. A salt of a strong base and a strong acid will yield a **neutral** aqueous solution.
      i. Example: NaCl (A salt from NaOH & HCl)
   b. A salt of a strong base and a weak acid will yield a **basic** solution.
      i. Example: NaCN (A salt from NaOH & HCN)
   c. A salt of a weak base and a strong acid will yield an **acidic** aqueous solution
      i. Example: $NH_4Cl$ (A salt from $NH_3$ & HCl)
   d. A salt of a weak base and a weak acid will depend on the relative acid – base strengths of the two ions.
      i. The $K_b$ and $K_a$ must be assessed to determine acidity or basicity.
         1. If the base is stronger than the acid ($K_b > K_a$), then the solution will be basic (pH > 7).
         2. If the acid is stronger than the base ($K_a > K_b$), then the solution will be acidic (pH < 7).
      ii. Example: $NH_4CHO_2$ (A salt from $NH_3$ & $HCHO_2$)
         1. $NH_4^+$: $K_a = 5.6 \times 10^{-10}$
         2. $CHO_2^-$: $K_b = 5.9 \times 10^{-11}$
      iii. This will be slightly acidic because $NH_4^+$ is a stronger acid than $HCO_2^-$ is a base.

**Introduction to Buffers:**
1) A **buffer** is a solution characterized by the ability to resist changes in pH when limited amounts of acids or bases are added to it.

  a. Buffers contain either a weak acid and its conjugate base or a weak base and its conjugate acid.
2) Blood is a buffer solution with a pH ranging from 7.35 – 7.45.
  a. The main conjugate acid–base pair is $H_2CO_3(aq)$ and $HCO_3^-(aq)$.
3) There are two important characteristics of buffers.
  **a.** The **Buffer capacity**
    i. This is the amount of acid or base that can react with the buffer before a significant pH change is reported.
  **b.** The **Buffer pH**
    i. Depending on the pH range of interest, the correct buffer must be selected to provide the optimal pH range.

## The Henderson – Hasselbalch Equation:

1) **The Henderson – Hasselbalch Equation** is an equation that determines pH of a buffer for different concentrations of the conjugate acid and base.
2) The equation is represented as:

$$pH = pK_a + \log \frac{[Base]}{[Acid]}$$

3) In any buffer, the weak acid and its conjugate base do not react appreciably with one another when the solutions are mixed.
  a. Thus, their relative concentrations can be calculated from the way the buffer is prepared.

## The Unknown Aqueous Salt Solutions:

1) KI (0.10 $M$)
2) $FeCl_3$ (0.10 $M$)
3) $Na_2CO_3$ (0.10 $M$)

## The Known Aqueous Solutions for Buffers:

1) $HC_2H_3O_2$ (0.1 $M$)
2) $NaC_2H_3O_2$ (0.1 $M$)

## Pre–Lab Information:

2) Provide the IUPAC names of KI, $FeCl_3$, and $Na_2CO_3$ and determine if aqueous solutions of each salt are acidic, basic, or neutral.
3) Obtain the true $pK_a$ value for acetic acid and provide a reference.

# Part 1:
## Experimental Procedure for Analysis of the Unknown Aqueous Salt Solutions:

1) Obtain 3 clean and completely dried test tubes, a 250 mL beaker, and one clean and dried 10.0 mL graduated cylinder.
2) Label each of the three test tubes by their unknown identity number/letter.
3) Place approximately 5.0 mL of each unknown salt solution into their labeled test tube.
4) Obtain a pH probe (or pH meter).

d. If advised, calibrate the pH probe with the buffer solutions provided.
5) Remove the pH probe from the electrode storage solution and rinse well with deionized/distilled water to achieve a pH of around 7.0.
    a. Do not discard electrode solution unless advised.
6) Soak the pH probe in deionized/distilled water in a 250 mL beaker.
    a. Record the pH value.
7) Remove pH from water soak and carefully dry off the probe with a Kimwipe.
8) Next, insert the pH probe into the solution and allow the for a stable pH value.
    d. Record this value in the data table.
9) Using deionized/distilled water, thoroughly rinse off the pH probe and then store in a 250 mL beaker of deionized water with the probe completely immersed.
10) Repeat steps 7–8 with all remaining solutions. Do not forget to rinse off probe well between readings and dry with a Kimwipe.
11) Calculate the pH, pOH, $[H_3O^+]$, and $[OH^-]$.
12) Determine the identity of each unknown salt solution.

## Part 2:
## Experimental Procedure for Formation and Analysis of Buffer Solutions:
1) Obtain five clean and completely dry 50–150 mL beakers, a glass stirring rod, one 250 mL beaker, and one clean and dried 10.0 mL and 50.0 mL graduated cylinder.
2) Obtain a pH probe (or pH meter).
    a. If advised, calibrate the pH probe with the buffer solutions provided.
3) Remove the pH probe from the electrode storage solution and rinse well with deionized/distilled water to achieve a pH of around 7.0.
    a. Do not discard electrode solution unless advised.
4) Soak the pH probe in deionized/distilled water in a 250 mL beaker.
    a. Record the pH value.
5) Add 30.0 mL of tap water to a 50.0 mL graduated cylinder and transfer into one of the 100 – 150 mL beakers.
6) Insert the pH probe into the water and allow the for a stable pH value.
    a. Record this value in the data table.
7) Add 5 drops of 0.10 $M$ HCl and stir thoroughly with the glass stirring rod.
8) Insert the pH probe into the solution and allow the for a stable pH value.
    a. Record this value in the data table.
9) Using deionized/distilled water, thoroughly rinse off the pH probe and then store in a 250 mL beaker of deionized water with the probe completely immersed.
10) Repeat the above procedure (steps 5–8) using 5 drops of 0.10 $M$ NaOH in place of the hydrochloric acid. Do not forget to rinse off probe and glass stirring rod and dry with a Kimwipe.
11) To a cleaned and dried 50.0 mL graduated cylinder, add 15.0 mL of the 0.10 $M$ acetic acid solution and transfer into one of the 100–150 mL beakers.
12) Rinse the graduated cylinder with deionized water and dry.
13) Add 15.0 mL of the 0.10 $M$ sodium acetate solution to the 50.0 mL graduated cylinder and transfer into the beaker containing the acetic acid solution.
14) Mix thoroughly with the glass stirring rod.
15) Insert the pH probe into the solution and allow the for a stable pH value.

a. Record this value in the data table.
b. Calculate the $[H_3O^+]$ and the experimental p$K_a$ for the acid.
16) Using deionized/distilled water, thoroughly rinse off the pH probe and then store in a 250 mL beaker of deionized/distilled water with the probe completely immersed.
17) Measure out 30.0 mL of tap water into the 50.0 mL graduated cylinder, transfer into the buffer solution, and mix thoroughly with the glass stirring rod.
18) Pour half of the new diluted buffer solution into another 100 – 150 mL beaker.
19) Insert the pH probe into one of the diluted buffer solutions and allow the for a stable pH value.
a. Record this value in the data table.
b. Calculate the $[H_3O^+]$ and the experimental p$K_a$ for the acid.
20) Add 5 drops of 0.10 M HCl to one beaker of the diluted buffer solution. Stir thoroughly with the glass stirring rod.
21) Add 5 drops of 0.1 M NaOH to the second beaker of the diluted buffer solution. Stir thoroughly with the glass stirring rod.
22) Measure the pH of each solution with the pH probe using the same procedure as indicated above.
a. Record the pH value in the data table
b. Calculate the $[H_3O^+]$ for each solution.
23) Create a buffer mixture of 2.0 mL of 0.10 *M* acetic acid solution and 20.0 mL of 0.10 *M* sodium acetate solution in a 100–150 mL beaker. Mix thoroughly with glass stirring rod.
24) Measure the pH of the solution with the pH probe using the same technique as described above.
a. Record the pH value in the data table
b. Calculate the $[H_3O^+]$ and p$K_a$ for the new buffer solution.
25) To the new buffer solution, add 3.0 mL of 0.10 M NaOH. Stir thoroughly with the glass stirring rod.
26) Measure the pH of the solution with the pH probe using the same technique as described above.
a. Record the pH value in the data table
b. Calculate the $[H_3O^+]$ for the new buffer solution.
27) Calculate an average experimental p$K_a$ value for acetic acid for all of the above trials.
28) Using this experimental p$K_a$ value, create a buffer system using the 0.10 *M* acetic acid and 0.10 *M* sodium acetate solutions that has a pH of 3.50.
29) Measure the pH of your solution with the pH probe using the same procedure as indicated above.
a. Record the pH value in the data table
b. Calculate the $[H_3O^+]$ for each solution.
30) Rinse pH probes thoroughly with deionized/distilled water. Dry with a Kimwipe and then return to the electrode storage solution.

## Waste Disposal and Lab Clean – Up:
1) All unknown aqueous salt solutions can be placed down the sink drain with plenty of water.
2) All buffer solutions can be neutralized with sodium hydroxide or sodium bicarbonate and then placed down the sink drain with plenty of water.

**The Lab Write – Up:**

1) Determine all values in your data table and discuss relevant data in the conclusion.
2) Predict whether aqueous solutions of KI, $FeCl_3$, and $Na_2CO_3$ are acidic, basic or neutral. Explain.
3) Show the solvation and hydrolysis reactions for each salt and place into your balanced equation section.
4) Determine the identity of all unknown aqueous salt solutions and thoroughly explain using pH data.
5) Discuss how the pH changed upon addition of 5 drops of HCl and NaOH with water.
6) Discuss how the pH changed upon addition of 5 drops of HCl and NaOH with all buffer solutions.
7) Compare and contrast the buffering ability of pure water with all of the acetic acid–sodium acetate buffers.
8) Determine the average experimental $pK_a$ value for acetic acid for the acetic acid–sodium acetate buffer. Compare to true value and discuss deviations.
9) Was the pH of your home–made buffer system close to 3.50? Explain deviations.
10) What assumptions were made during this experiment?

# Pre–Lab Data Tables:

| Formula of Salt | IUPAC Name | Acid, Base, or Neutral |
|---|---|---|
| KI | | |
| $FeCl_3$ | | |
| $Na_2CO_3$ | | |

True/Theoretical p$K_a$ value for acetic acid: _____

Reference: _____

_____

# Balanced Equations:

| Process and Salt | Reactant(s) | Yields | Product(s) |
|---|---|---|---|
| Solvation for KI | ___ KI(s) | $\xrightarrow{H_2O}$ | |
| Hydrolysis of KI ions | | $\rightleftharpoons$ | |
| | | | |
| Solvation for $FeCl_3$ | ___ $FeCl_3$(s) | $\xrightarrow{H_2O}$ | |
| Hydrolysis of $FeCl_3$ ions | | $\rightleftharpoons$ | |
| | | | |
| Solvation for $Na_2CO_3$ | ___ $Na_2CO_3$(s) | $\xrightarrow{H_2O}$ | |
| Hydrolysis of $Na_2CO_3$ ions | | $\rightleftharpoons$ | |

## Sample Data Table for Part 1:

| Aqueous Salt Solution # | pH | pOH | $[H_3O^+]$ | $[OH^-]$ | Identity of Salt Solution |
|---|---|---|---|---|---|
| 1 (A) | | | | | |
| 2 (B) | | | | | |
| 3 (C) | | | | | |

## Sample Data Table for Part 2:

| Solution | 0.10 $M$ HCl | 0.10 $M$ NaOH | pH | $[H_3O^+]$ | Experimental $pK_a$ |
|---|---|---|---|---|---|
| 30.0 mL tap water | 0 | 0 | | | N/A |
| 30.0 mL tap water | 5 drops | 0 | | | N/A |
| 30.0 mL tap water | 0 | 5 drops | | | N/A |
| 15.0 mL 0.10 $M$ acetic acid + 15.0 mL 0.10 $M$ sodium acetate | 0 | 0 | | | |
| 15.0 mL 0.10 $M$ acetic acid + 15.0 mL 0.10 $M$ sodium acetate + 30.0 mL tap water | 0 | 0 | | | |
| 15.0 mL 0.10 $M$ acetic acid + 15.0 mL 0.10 $M$ sodium acetate + 30.0 mL tap water (Beaker #1 – Half) | 5 drops | 0 | | | N/A |
| 15.0 mL 0.10 $M$ acetic acid + 15.0 mL 0.10 $M$ sodium acetate + 30.0 mL tap water (Beaker #2 – Half) | 0 | 5 drops | | | N/A |
| 2.0 mL 0.10 $M$ acetic acid + 20.0 mL 0.10 $M$ sodium acetate | 0 | 0 | | | |
| 2.0 mL 0.10 $M$ acetic acid + 20.0 mL 0.10 $M$ sodium acetate | 0 | 3.0 mL | | | N/A |
| Your Solution: _____ _____ | N/A | N/A | | | Average $pK_a$: _____ |

# Experiment #9:
# The Analysis of the Solubility of Calcium Hydroxide and Determination of the Solubility Product Constant

## Introduction to Solubility Equilibria:
1) Many natural processes rely upon the dissolution or precipitation of slightly soluble salts.
2) The blood contains a unique balance of slightly or sparingly soluble salts.
   a. Examples:
      i. Calcium phosphate: $Ca_3(PO_4)_2$
      ii. Calcium oxalate: $CaC_2O_4$
      iii. Magnesium carbonate: $MgCO_3$
      iv. Magnesium oxalate: $MgC_2O_4$
3) When an excess of a slightly soluble ionic compound is mixed with water, an equilibrium is established between the solid and the ions in the saturated solution.
   a. When calcium oxalate is dissolved in water, the following solubility equilibrium is achieved:

$$CaC_2O_4(s) \xrightleftharpoons{\text{H}_2\text{O}} Ca^{2+}(aq) + C_2O_4^{2-}(aq)$$

   i. (above)
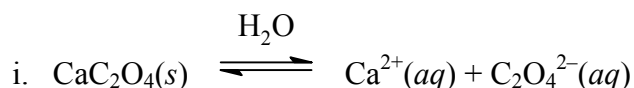
## Introduction to the Solubility Product ($K_{sp}$):
1) Generally, the solubility product constant ($K_{sp}$) is the equilibrium constant for the solubility equilibrium of a slightly soluble (or nearly insoluble) ionic compound.
2) The $K_{sp}$ is the product of the equilibrium concentrations of the ions in the compound at a certain temperature.
   a. Like reaction equilibrium constants, the solubility product constants are dependent on temperature.
3) Each concentration is raised to a power equal to the number of such ions in the formula of the compound.
4) The solubility product for calcium oxalate is as follows:

$$K_{sp} = [Ca^{2+}][C_2O_4^{2-}]$$

## Introduction to the Ion Product Quotient ($Q_{sp}$):
1) Precipitation reactions can ensue by a disturbance in the solubility equilibrium.
2) To determine whether an equilibrium system will go in the forward or reverse direction resulting in a precipitation reaction, it requires the evaluation of the ion product quotient, $Q_{sp}$.
3) To predict the direction of reaction, compare the values of $Q_{sp}$ with $K_{sp}$.
   a. If $Q_{sp}$ exceeds the $K_{sp}$, precipitation occurs.
   b. If $Q_{sp}$ is less than $K_{sp}$, more solute can dissolve (and precipitation does not occur.)
   c. If $Q_{sp}$ equals the $K_{sp}$, the solution is saturated.

## The Solubility of Calcium Hydroxide:

1) Although calcium hydroxide, $Ca(OH)_2$, is a strong base, it is only slightly soluble in water.
2) When calcium hydroxide is dissolved in water, the following solubility equilibrium is achieved:

   a. $Ca(OH)_2(s) \xrightleftharpoons{H_2O} Ca^{2+}(aq) + 2OH^-(aq)$

3) The solubility expression for calcium hydroxide is as follows: $K_{sp} = [Ca^{2+}][OH^-]^2$
4) This experiment will be a two – step process.
   a. In the first part, the solubility of a solution of calcium hydroxide will be "challenged" by introduction of a common ion and temperature changes.
   b. In the second part, a saturated solution of calcium hydroxide will be titrated with a strong acid to determine the solubility product constant ($K_{sp}$).

## Pre–Lab Information:

1) Obtain the true values for the solubility (g/L) at $0^oC$ and the solubility product constant ($K_{sp}$) at $25^oC$ for calcium hydroxide. Provide a reference.

# Part 1(a):
## Procedure for the Temperature Challenge:

1) Obtain a clean and completely dried 100.0 mL volumetric flask, a 250 mL Erlenmeyer flask, a glass stirring rod (and/or a magnetic stir bar), a thermometer or a temperature probe, a Büchner funnel with filter paper, a filter flask with vacuum adapter and vacuum tubing, a 400–600 mL beaker, and a hot plate.
2) Create an ice–water bath with the 400–600 mL beaker that is large enough to accommodate the 250 mL Erlenmeyer flask.
3) Using a wax pencil, label the 250 mL Erlemeyer flask as "Temp."
4) Using weighing paper, weigh out 0.125 g of calcium hydroxide on the analytical balance.
   a. Record the exact mass of each in the laboratory notebook.
5) Place the calcium hydroxide into a 100.0 mL volumetric flask and at **up to** 100 mL of water.
6) Agitate for a few minutes and ensure that the majority of the powder has dissolved.
   a. Assume that the entire mass of calcium hydroxide powder measured out dissolved in water to calculate the molarity.
7) Set up vacuum filtration system using the Büchner funnel.
   a. See diagram attached.
   b. After turning on vacuum, wet the filter paper used with deionized/distilled water.
8) Vacuum filter the calcium hydroxide solution and then transfer filtrate into the 250 mL Erlenmeyer flask labeled "Temp.".
   a. Ensure that the solution is completely clear. Re–filter if necessary.
9) Record the initial temperature of the calcium hydroxide solution labeled "Temp" with a thermometer or a temperature probe.
   a. Also, record color and clarity of solution.

10) Immerse the solution into the ice–water bath and allow the temperature to decrease to approximately 0.0°C.
    a. Record a stable temperature between 0–10°C and the solubility observations.
11) Remove solution from the ice–water bath and dry off exterior of the beaker.
12) Clamp the Erlenmeyer flask labeled "Temp" into a water bath on a hot plate and increase the temperature of the solution to greater than 70°C.
    a. Record a stable temperature >70°C and the solubility observations.

## Part 1(b):

### Procedure for the Ion Challenge:

1) Obtain a clean and completely dried 100 mL volumetric flask, a 250 mL Erlenmeyer flask, a 100 mL graduated cylinder, a glass stirring rod (and/or a magnetic stir bar), a thermometer or a temperature probe, a Büchner funnel with filter paper, a filter flask with vacuum adapter and vacuum tubing, a 150 mL beaker, a 400–600 mL beaker, and a hot plate.
2) Using a wax/grease pencil, label the 250 mL Erlemeyer flask as "Ion."
3) Using weighing paper, weigh out 0.125 g of calcium hydroxide on the analytical balance.
    a. Record the exact mass of each in the laboratory notebook.
4) Place the calcium hydroxide into a 100.0 mL volumetric flask and at **up to** 100 mL of water.
5) Agitate for a few minutes and ensure that the majority of the powder has dissolved.
6) Set up vacuum filtration system using the Büchner funnel.
    a. See diagram attached.
    b. After turning on vacuum, wet the filter paper used with deionized/distilled water.
7) Vacuum filter the calcium hydroxide solution and then transfer filtrate into the 250 mL Erlenmeyer flask labeled "Ion".
    a. Ensure that the solution is completely clear. Re–filter if necessary.
8) Record the initial temperature of the calcium hydroxide solution labeled "Ion" with a thermometer or a temperature probe.
    a. Also, record color and clarity of solution.
9) Measure out 100.0 mL of 6.0 $M$ NaOH in a graduated cylinder and transfer into a 150 mL beaker. Label as "NaOH."
10) Determine if a precipitation reaction will take place **if** the sodium hydroxide solution were added to the calcium hydroxide solution.
    a. This can be determined by calculating the $Q_{sp}$ for the mixture and comparing to the known $K_{sp}$ value for calcium hydroxide.
        i. Assume that the entire mass of calcium hydroxide powder measured out dissolved in water to calculate the molarity.
    b. Perform this calculation BEFORE performing the next step.
11) Transfer the 100 mL of 6.0 M NaOH to the Erlenmeyer flask labeled "Ion" and mix well with the glass stirring rod.
    a. Record the solubility observations.

# Part 2:
## Procedure for Calculation of the $K_{sp}$ of Calcium Hydroxide:

1) Obtain two clean and completely dry 400 mL beakers, a buret with stand, a hot plate with a stir function, a magnetic stir bar, one clean and dried 100 mL graduated cylinder, a thermometer or a temperature probe, one 250 mL Erlenmeyer flask, a Büchner funnel with filter paper, a filter flask with vacuum adapter and vacuum tubing, one 250 mL beaker, a short-stem funnel, a utility clamp, and a glass stirring rod.
2) Obtain a pH probe (or pH meter).
   e. If advised, calibrate the pH probe with the buffer solutions provided.
3) Remove the pH probe from the electrode storage solution and rinse well with deionized/distilled water to achieve a pH of around 7.0.
   b. Do not discard electrode solution unless advised.
4) Soak the pH probe in deionized/distilled water in a 250 mL beaker.
   a. Record the pH value.
5) Obtain approximately 225 mL of a saturated calcium hydroxide suspension in a 250 mL beaker.
6) Set up vacuum filtration system using the Büchner funnel.
   a. See diagram attached.
   b. After turning on vacuum, wet the filter paper used with deionized/distilled water.
7) Vacuum filter the saturated calcium hydroxide solution and then transfer filtrate into a 250 mL Erlenmeyer flask.
   a. Ensure that the solution is completely clear. Re–filter if necessary.
8) Using a 100.0 mL graduated cylinder, transfer exactly 100.0 mL of saturated calcium hydroxide solution into the first 400 mL beaker.
   a. With a wax/grease pencil, label as "Titration beaker #1."
9) Add 10 drops of phenolphthalein indicator to the beaker.
   a. Record the initial color and clarity of this solution.
10) Insert the magnetic stir bar into the solution and place onto the hot plate.
    a. Turn on the stir function and allow for a slow to moderate mixing.
    b. You will NOT need the heating function for the hot plate during this part.
11) Obtain approximately 100.0 mL of a standard 0.10 $M$ HCl solution into a dry 100 mL beaker.
    a. Label beaker with "standard HCl" with a wax/grease pencil.
12) Ensure the buret stopcock is in the closed position. Using a short stem funnel, add approximately 10.0 mL of the standardized HCl solution to the buret.
13) Condition the buret by holding it horizontally and rotating to allow the HCl solution to wet the inside of the glass completely. Discard the rinse into a properly labeled "Acid Waste" container.
14) Fill the buret with the standardized HCl solution and adjust the meniscus to zero.
    a. Record the initial buret reading as 0.00 mL.
    b. Keep any remaining HCl solution for later.
15) Carefully dry off the pH probe with a Kimwipe.
16) Next, using a utility clamp, suspend the pH probe to one side of the beaker into the calcium hydroxide solution and allow the for a stable pH value.
    e. Record this value in the data table.
    a. DO NOT allow the pH probe to come into contact with the magnetic stir bar.

17) Suspend the buret over the calcium hydroxide solution to the side opposite the pH probe.
    a. Upon delivering the standardized HCl solution, it is critical that the pH probe test the solution and not the dripping acid!
18) Record the initial temperature of the mixture with a thermometer and periodically throughout the addition of the HCl solution.
19) Add approximately 1.00 mL of the HCl solution and allow the pH to stabilize.
    a. Record the following information now and after each subsequent addition of HCl solution:
        i. The volume of HCl solution delivered via the buret.
        ii. The pH value once stabilized.
        iii. Observations, including the temperature and color/clarity of the solution.
20) Continue to add 1.00 mL of the HCl solution until the change of pH is greater than 0.50 from the previous reading.
    a. Note the deep pink color should begin to fade throughout the beginning part of this titration.
    b. The colorimetric endpoint of the titration is when the addition of a single added drop of HCl solution produces a completely colorless solution. Try to capture this endpoint.
    c. Since a strong base is being titrated with a strong acid, the equivalence point (as recorded via the pH probe) should occur at a pH of 7.00.
21) When the change of pH is greater than 0.50 from the previous reading, begin adding only 0.25–0.50 mL increments of the HCl solution.
    a. You may need to add smaller volumes, when necessary, to capture the endpoint.
22) Continue adding these smaller volumes (0.25–0.50 mL) of HCl solution until the change of pH is less than 0.50 from the previous value.
23) Once the endpoint has been reached, add at least 5.0 mL more of the HCl solution in 0.50–1.00 mL increments.
24) Repeat the above procedure with another 100 mL of saturated calcium hydroxide solution to ensure reproducibility.
25) Remove the pH probe and rinse thoroughly with deionized/distilled water. Dry with a Kimwipe and then return to the electrode storage solution.
26) Remove the HCl solution from the buret and neutralize as discussed below. Carefully clean the buret thoroughly.
27) Plot a titration curve and determine the equivalence point.
    a. Place the volume of HCl on the x–axis and pH on the y–axis.
    b. Compare the equivalence point to the endpoint.
28) Calculate the $K_{sp}$ of calcium hydroxide and compare to the true value.


## Waste Disposal and Lab Clean–Up:
1) Place all calcium hydroxide and hydrochloric acid solutions into the same waste beaker. If neutral or slightly basic, flush down the sink with plenty of water.
2) Unused or waste HCl solution can be neutralized with the calcium hydroxide solution. If neutral or slightly basic, flush down the sink with plenty of water.

**The Lab Write – Up**:

1) Determine all values in your data table and discuss relevant data in your conclusion.
2) For the first part of the experiment:
   a. Discuss the temperature dependence on the solubility of calcium hydroxide.
   b. Discuss the expected solubility of calcium hydroxide in a solution of sodium hydroxide comparing the calculated $Q_{sp}$ to the theoretical $K_{sp}$.
3) For the second part of the experiment:
   a. Plot a titration curve and determine the equivalence point.
   b. Determine the value for $K_{sp}$ and the solubility (in g/L) for this reaction at room temperature.
   c. Compare to the true values and discuss discrepancies.
4) Compare and contrast the colorimetric endpoint (via the phenolphthalein indicator) with the equivalence point (via pH data).
5) Show the balanced equation from the reaction between calcium hydroxide with hydrochloric acid and discuss how the **solubility** equilibrium for $Ca(OH)_2$ is shifted.
6) Discuss all sources of error during this experiment and how the data is directly affected.


# Pre–Lab Information:

True $K_{sp}$ of $Ca(OH)_2$ @ Temperature: _____ @ $25^{\circ}C$

Reference: _____

True Solubility of $Ca(OH)_2$ (g/L) @ Temperature: _____ @ $0^{\circ}C$

Reference: _____

# Sample Data Tables:

Part 1(a): Temperature Challenge

Mass of $Ca(OH)_2$ (g): _____

Moles of $Ca(OH)_2$ (mol): _____

Total volume of solution (mL): <u>100.0 mL (0.1000 L)</u>

Calculated molarity of $Ca(OH)_2$ (mol/L): _____

| Temperature ($^o$C) | Solubility Observations (Color/Clarity) |
|---|---|
| Initial: _____ | |
| Cold: _____ | |
| Hot: _____ | |

Part 1(b): Common Ion Challenge:

Mass of $Ca(OH)_2$ (g): _____

Moles of $Ca(OH)_2$ (mol): _____

Total volume of solution (mL): <u>100.0 mL (0.1000 L)</u>

Calculated molarity of $Ca(OH)_2$ (mol/L): _____

Initial temperature ($^o$C): _____

Volume of 6.0 $M$ NaOH (mL): _____

Calculated $Q_{sp}$ for $Ca(OH)_2$ + NaOH: _____

Will a precipitation reaction occur? (Yes/No) _____

Solubility observations following mixing: _____

# Titration of Saturated Ca(OH)$_2$ Solution – Trial #1

| pH | Volume of HCl Added (mL) | Observations (Temperature and Color/Clarity) |
|---|---|---|
| | 0.00 | |
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# Titration of Saturated Ca(OH)₂ Solution – Trial #2

| pH | Volume of HCl Added (mL) | Observations (Temperature and Color/Clarity) |
|---|---|---|
|  | 0.00 |  |
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# Sample Data Tables Continued:

| Data Recorded/Calculated | Trial #1 | Trial #2 |
|---|---|---|
| a) Standardized molar concentration of HCl used in titration in moles per liter: | | |
| | | |
| b) Volume of saturated calcium hydroxide solution titrated in milliliters: | 100.0 mL | 100.0 mL |
| | | |
| c) Endpoint volume of HCl solution **via phenolphthalein indicator** in milliliters: (See titration observations) | | |
| d) Equivalence point volume of HCl solution **via pH data** in milliliters: (See titration curves) | | |
| e) Average equivalence point volume of HCl solution **via pH data** in milliliters: | | |
| | | |
| f) Average moles of HCl solution used to reach equivalence point in mol[*]: | | |
| | | |
| g) Average moles of $OH^-$ upon titration to equivalence point in mol[†]: | | |
| h) Average moles of $Ca^{2+}$ upon titration to equivalence point in mol[††]: | | |
| | | |
| i) Average molarity of $OH^-$ upon titration to equivalence point in mol per liter: | | |
| j) Average molarity of $Ca^{2+}$ upon titration to equivalence point in mol per liter: | | |
| | | |
| k) Experimental $K_{sp}$ for calcium hydroxide @ temperature in degrees Celsius[†††]: | | _____ @ _____ °C |
| k) True $K_{sp}$ for calcium hydroxide @ temperature in degrees Celsius[†††]: | | _____ @ _____ °C |

> [*]Using standardized molarity of HCl in data point a)
> [†]Using mole to mole ratio of HCl to $OH^-$
> [††]Using: 1 mol $OH^- = \frac{1}{2}$ mol $Ca^{2+}$
> [†††]Using $K_{sp} = [Ca^{2+}][OH^-]^2$

# Model of Vacuum Filtration Using a Büchner Funnel
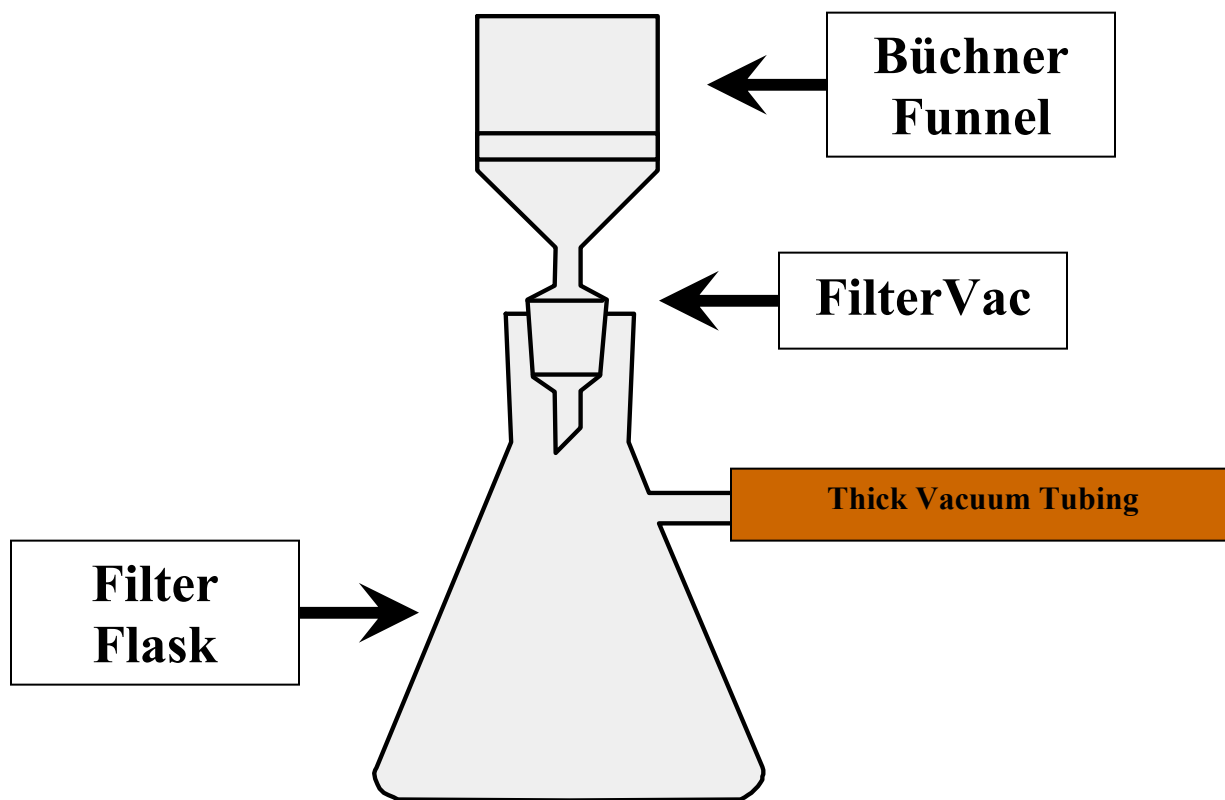
**Büchner Funnel**

**FilterVac**

**Thick Vacuum Tubing**

**Filter Flask**

**Clamp filter flask securely to a ring stand before affixing vacuum tubing and turning on vacuum.**

<div style="border: 2px solid red;">

# Experiment #10:
# Transition Metal Complexes; A Comparison of Formation Constants

</div>

## Introduction to Complex – Ion Equilibria:

1) Many metal ions, especially transition metals, form coordinate covalent bonds with molecules or anions having a lone pair of electrons.
   a. This type of bond formation is essentially a Lewis acid–base reaction.
   b. Example: $Ag^+(aq) + 4\,SCN^-(aq) \rightleftharpoons [Ag(SCN)_4]^{3-}(aq)$
      i. The silver ion is the Lewis acid (electron pair acceptor) and thiocyanate ion is the Lewis base (electron pair donor.)
2) A complex is defined as a compound containing complex ions.
3) A ligand is a Lewis base (an electron pair donor) that bonds to a metal ion to form a complex ion.
4) The **formation constant, $K_f$,** is the equilibrium constant for the formation of a complex ion from the aqueous metal ion and the ligands.
   a. The formation constant for $[Ag(SCN)_4]^{3-}(aq)$ is as follows: $K_f = \dfrac{[[Ag(SCN)_4]^{3-}]}{[Ag^+][SCN^-]^4}$
   b. The value for $K_f$ for $[Ag(SCN)_4]^{3-}(aq)$ is $1.2 \times 10^{10}$.
   c. A large value for $K_f$ means that the complex ion is quite stable.
5) The **dissociation constant, $K_d$,** is the reciprocal, or inverse, value of $K_f$.
   a. The dissociation constant for $[Ag(SCN)_4]^{3-}(aq)$ is as follows: $K_d = \dfrac{1}{K_f} = \dfrac{[Ag^+][SCN^-]^4}{[[Ag(SCN)_4]^{3-}]}$
   b. The value for $K_d$ for $[Ag(SCN)_4]^{3-}(aq)$ is $8.3 \times 10^{-11}$.

## The Reagents:

1) $0.10\ M$ $AgNO_3$
2) $0.10\ M$ $Cu(NO_3)_2$
3) $0.10\ M$ $Ni(NO_3)_2$
4) $6.0\ M$ $NH_4OH$ (source of $NH_3$)

## Pre–Lab Information:

1) In preparation for the laboratory experiment, complete "Table 1: $K_f$ and $K_d$ Values of the Transition Metal Ammonia Complexes" in advance of the lab period and properly reference the source(s) of information.

## Part 1–Experimental Procedure for Formation of Ammonia Complexes:

1) Obtain 6 test tubes with a test tube rack, a glass stir rod, a 10.0 mL graduated cylinder, a wax/grease pencil, and a deionized/distilled water bottle.
2) Using the wax/grease pencil, label the first three test tubes as "$Ag^+$", "$Cu^{2+}$", and "$Ni^{2+}$".
3) Using a 10.0 mL graduated cylinder, add $8 - 10$ mL of $0.10\ M$ $AgNO_3$ into the test tube labeled "$Ag^+$".
   a. Record the color and clarity of the solution.
4) Add $8 - 10$ mL of $0.10\ M$ $Cu(NO_3)_2$ and $0.10\ M$ $Ni(NO_3)_2$ to the properly labeled test tubes.
   a. Record the color and clarity of the solutions.

b. It may be advisable to only use the graduated cylinder to measure out the first solution and "eyeball" the correct amount of liquid to fill the other test tubes. This will prevent contamination.
5) To each solution separately, add 10 – 20 drops of deionized/distilled water.
   a. Stir with a glass stir rod.
   b. Record the color and clarity of the solutions.
   c. Note: Clean and dry glass stir rod well before using in each solution.
6) Next, to each solution separately, add 10 – 20 drops of 6.0 $M$ $NH_4OH$ (source of $NH_3$).
   a. Stir with a glass stir rod.
   b. Record the color and clarity of the solutions.
   c. Note: Clean and dry glass stir rod well before using in each solution.
7) Lastly, to each solution separately, add 10 – 20 drops of deionized/distilled water.
   a. Stir with a glass stir rod.
   b. Record the color and clarity of the solutions.
   c. If no real change is observed, this means that the ammonia complexes have formed and are more stable than the aqua (water) complexes.
8) Do not discard the three ammonia complexes as they will be used in part 2.

## Part 2–Experimental Procedure for Testing Relative Stability of Ammonia Complexes:
1) Using the wax/grease pencil, label the second three test tubes as "$Ag^+$", "$Cu^{2+}$", and "$Ni^{2+}$".
2) Using the three formed ammonia complexes in part 1, pour half of the solution into the properly labeled test tube.
   a. You should now have 2 test tubes of each ammonia complex.
   b. Record the initial color and clarity of the solutions.
3) To one of the test tubes labeled "$Ag^+$", add 10 – 20 drops of 0.10 $M$ $Cu(NO_3)_2$.
   a. Stir with a glass stir rod.
   b. Record the color and clarity of the solution.
   c. Note: Clean and dry glass stir rod well before using in each solution.
4) Repeat step 3 using 10 – 20 drops of 0.10 $M$ $Ni(NO_3)_2$.
5) To one of the test tubes labeled "$Cu^{2+}$", add 10 – 20 drops of 0.10 $M$ $AgNO_3$.
   a. Stir with a glass stir rod.
   b. Record the color and clarity of the solution.
   c. Note: Clean and dry glass stir rod well before using in each solution.
6) Repeat step 5 using 10 – 20 drops of 0.10 $M$ $Ni(NO_3)_2$.
7) To one of the test tubes labeled "$Ni^{2+}$", add 10 – 20 drops of 0.10 $M$ $AgNO_3$.
   a. Stir with a glass stir rod.
   b. Record the color and clarity of the solution.
   c. Note: Clean and dry glass stir rod well before using in each solution.
8) Repeat step 7 using 10 – 20 drops of 0.10 $M$ $Cu(NO_3)_2$.
9) Compare the observations recorded to the $K_f$ values to assess if there is a correlation.

## Waste Disposal and Lab Clean – Up:
1) Place all solutions into the same properly labeled waste container.

**The Lab Write – Up**:

1) Determine all values in your data table and discuss relevant data in your conclusion.
2) In the conclusion, discuss how $K_f$ and $K_d$ relate to the stability of a complex ion.
    a. Provide the equilibrium constant expressions for the ammonia complexes analyzed.
    b. Provide the values for $K_f$ and $K_d$ for the ammonia complexes analyzed.
3) In the conclusion, compare the observations recorded to the $K_f$ values to assess if there is a correlation.
4) Provide an appropriate balanced equation for each ammonia complex formation.
5) Discuss all sources of error during this experiment and how these errors directly affected the data.

# Pre – Lab Information:

## Table 1: $K_f$ and $K_d$ Values of the Transition Metal Ammonia Complexes:

| Heavy Metal Ammonia Complex | $K_f$* | $K_d$ | Stability Ranking (1 = most, 3 = least) |
|---|---|---|---|
| $[Ag(NH_3)_2]^+(aq)$ | | | |
| $[Cu(NH_3)_4]^{2+}(aq)$ | | | |
| $[Ni(NH_3)_6]^{2+}(aq)$ | | | |

*Provide reference: _____

## The Balanced Equations:

| Reactant(s) | | Product(s) |
|---|---|---|
| ___ $Ag^+(aq)$ + ___ $NH_3(aq)$ | ⇌ | |
| ___ $Cu^{2+}(aq)$ + ___ $NH_3(aq)$ | ⇌ | |
| ___ $Ni^{2+}(aq)$ + ___ $NH_3(aq)$ | ⇌ | |
| ___ $NH_4OH(aq)$* | ⇌ | |

**\*Side reaction**

# Sample Data Tables:

## Part 1: Formation of Ammonia Complexes

| Heavy Metal Solution | Initial Color & Clarity | Color & Clarity After Water Addition | Color & Clarity After Ammonia Addition | Color & Clarity After Second Water Addition |
|---|---|---|---|---|
| 0.10 *M* AgNO$_3$(*aq*) | | | | |
| 0.10 *M* Cu(NO$_3$)$_2$(*aq*) | | | | |
| 0.10 *M* Ni(NO$_3$)$_2$(*aq*) | | | | |

## Part 2: Testing Relative Stability of Ammonia Complexes

| Heavy Metal Ammonia Complex | Initial Color & Clarity | Color & Clarity After 0.10 *M* AgNO$_3$(*aq*) Addition | Color & Clarity After 0.10 *M* Cu(NO$_3$)$_2$(*aq*) Addition | Color & Clarity After 0.10 *M* Ni(NO$_3$)$_2$(*aq*) Addition |
|---|---|---|---|---|
| [Ag(NH$_3$)$_2$]$^+$(*aq*) | | ✕ | | |
| [Cu(NH$_3$)$_4$]$^{2+}$(*aq*) | | | ✕ | |
| [Ni(NH$_3$)$_6$]$^{2+}$(*aq*) | | | | ✕ |

## An Introduction to Thermodynamics and Enthalpy (H):

1) Thermodynamics is the study of the relationship between heat and other forms of energy in a chemical or physical process.
2) The heat absorbed or evolved by a physical or chemical process depends on the conditions under which it occurs.
   a. Most processes take place in an open container, and thus are at constant pressure.
      i. This change in heat at constant pressure is denoted as $q_p$.
3) Enthalpy, denoted H, is an extensive property of a substance that can be used to obtain the heat absorbed or evolved in a physical or chemical process.
   a. Enthalpy is an extensive property of matter; it depends on the amount of a substance.
   b. Enthalpy is a state function which means that it is a property of a system that only depends on the present state and is independent of the previous history of that system.
4) The change in enthalpy ($\Delta H$) for a reaction at a given temperature and pressure (called the enthalpy of reaction) is obtained by subtracting the enthalpy of the reactants from the enthalpy of the products.
5) The term standard state refers to the standard thermodynamic conditions chosen for substances when listing or comparing thermodynamic data; this implies $25^{\circ}C$, 1 atm pressure, and a concentration of 1 $M$ for dissolved substances.
   a. A standard enthalpy is denoted as $\Delta H^{o}$.
6) The standard enthalpy of formation of a substance, denoted $\Delta H_f^{o}$, is the enthalpy change for the formation of one mole of a substance in its standard state from its component elements in their standard state.
   a. The standard enthalpy of formation for elements is always equal to zero kilojoules per mole.
7) The law of summation of heats of formation states that the enthalpy of a reaction is equal to the total formation energy of the products minus that of the reactants.
   a. The equation: $\Delta H^{o} = \sum n\Delta H_f^{o}(\text{products}) - \sum m\Delta H_f^{o}(\text{reactants})$
   b. The letters $m$ & $n$ are coefficients for those substances in the balanced chemical equation.

## An Introduction to Entropy (S):

1) The second law of thermodynamics states that for a totally isolated system that is not at thermal equilibrium, the entropy will increase over time reaching a maximum.
   a. Entropy, S, is a thermodynamic quantity that is a measure of the randomness or disorder of a system.
   b. The SI unit of entropy is joules per Kelvin (J/K) and, like enthalpy, is a state function.

2) The third law of thermodynamics states that a substance that is perfectly crystalline at a temperature of 0 Kelvin has an entropy of zero joules per Kelvin.
   a. When temperature is raised, however, the substance becomes more disordered as it absorbs heat.
   b. The entropy of a substance is determined by measuring how much heat is required to change its temperature per Kelvin degree.
3) The entropy change for a reaction can be calculated by using a summation law, similar to the way $\Delta H_f^o$ is obtained.
   a. The equation: $\Delta S^o = \sum n\Delta S^o(\text{products}) - \sum m\Delta S^o(\text{reactants})$
4) The entropy usually increases in the following situations:
   a. A reaction in which a molecule is broken into two or more smaller molecules.
   b. A reaction in which there is an increase in the moles of gases.
   c. A process in which a solid changes to liquid or gas or a liquid changes to gas

## An Introduction to Gibbs Free Energy (G):
1) The American physicist by the name of J. Willard Gibbs introduced the concept of free energy (sometimes called the Gibbs free energy), G.
2) Gibbs free energy is a measurement of the thermodynamic potential for maximum work energy at constant temperature and pressure.
   a. This quantity gives a direct criterion for spontaneity of reaction.
3) Changes in enthalpy ($\Delta H$) and entropy ($\Delta S$) during a reaction result in a change in free energy, $\Delta G$, given by the equation: $\Delta G = \Delta H - T\Delta S$
4) Gibbs free energy can be useful in judging the spontaneity of a reaction:
   a. When $\Delta G^o$ is a large negative number (more negative than about –10 kJ), the reaction is spontaneous as written, and the reactants transform almost entirely to products when equilibrium is reached.
   b. When $\Delta G^o$ is a large positive number (more positive than about +10 kJ), the reaction is nonspontaneous as written, and reactants do not give significant amounts of product at equilibrium.
   c. When $\Delta G^o$ is a small negative or positive value (less than about 10 kJ), the reaction gives an equilibrium mixture with significant amounts of both reactants and products.
5) The table below can be used to predict the spontaneity of a reaction given temperature considerations.

| $\Delta H^o$ | $\Delta S^o$ | $\Delta G^o$ | Description |
|:---:|:---:|:---:|:---:|
| – | + | – | Spontaneous at all temperatures |
| + | – | + | Nonspontaneous at all temperatures |
| – | – | + or – | Spontaneous at low temperatures Nonspontaneous at high temperatures |
| + | + | + or – | Spontaneous at high temperatures Nonspontaneous at low temperatures |

## The Reactions to Assess for Spontaneity at 25$^o$C:
1) $Cu(s) + 2H^+(aq) \rightarrow Cu^{2+}(aq) + H_2(g)$
2) $Mg(s) + 2H^+(aq) \rightarrow Mg^{2+}(aq) + H_2(g)$
3) $Mg(s) + 2\ H_2O(l) \rightarrow Mg(OH)_2(s) + H_2(g)$
4) $Ca(s) + 2\ H_2O(l) \rightarrow Ca^{2+}(aq) + 2\ OH^-(aq) + H_2(g)$

## Pre–Lab Information:
1) Fill out "Table #1: Standard Enthalpy and Entropy Data" by obtaining the values for standard enthalpies of formation ($\Delta H_f^o$) and standard entropies ($S^o$) for all substances in this table. Provide reference(s).

## Experimental Procedure:
1) Before performing each reaction, use the pre–lab data collected to calculate the $\Delta H^o$, $\Delta S^o$, $\Delta G^o$, and make a prediction as to the spontaneity of the reaction.
2) Obtain four clean and **completely** dry 16 x 150 mm test tubes in a test tube rack, a thermometer, a 10.0 mL graduated cylinder, wide-range pH paper, a wax/grease pencil, and a bottle of deionized/distilled water.
3) Using a wax/grease pencil, label each test tube as to the reaction # to be performed.
4) Using a 10.0 mL graduated cylinder, add 2–3 mL of 1.0 *M* HCl to test tube #1 and test tube #2.
5) Add 2–3 mL of deionized/distilled water into test tube #3 and test tube #4 using the squeeze bottle.
   a. You do not need to use a graduated cylinder for this step; fill these two test tubes to the same height as test tubes #1 and #2.
6) Insert the thermometer into test tube #1.
   a. Record the initial temperature of the liquid.
7) Acquire a 1 cm strip of copper metal.
   a. Collect observations as to physical properties of the metal such as color, luster, etc.
8) Temporarily remove the thermometer and then add the 1 cm strip of copper metal into test tube #1.
   a. Reinsert the thermometer immediately after the metal was added and monitor for a temperature change.
9) Allow to react for 5 minutes and collect reaction observations.
   a. Once 5 minutes has elapsed, record the final temperature of the liquid.
   b. If no reaction occurs, state that no changes were observed.
   c. Clean and dry thermometer well before moving to the next test tube.
10) Repeat steps 6 – 9 in test tube #2 using a 1 cm strip of magnesium metal.
    a. Make sure the magnesium metal has been polished well with sand paper or steel wool.
11) Insert the thermometer into test tube #3.
    a. Record the initial temperature of the liquid.
12) Using the thermometer, place a drop of the liquid onto a piece of pH paper.

      a.  Record the initial pH of the liquid.
13) Acquire a 1 cm strip of magnesium metal.
      a.  Make sure the magnesium metal has been polished well with sand paper or steel wool.
      b.  Collect observations as to physical properties of the metal such as color, luster, etc.
14) Temporarily remove the thermometer and then add the 1 cm strip of magnesium metal into test tube #3.
      a.  Reinsert the thermometer immediately after the metal was added and monitor for a temperature change.
15) Allow to react for 5 minutes and collect reaction observations.
      a.  Once 5 minutes has elapsed, record the final temperature of the liquid.
      b.  Use a glass stir rod to test pH of the resultant solution via the pH paper.  Compare the pH to pure deionized/distilled water.
      c.  If no reaction occurs, state that no changes were observed.
      d.  Clean and dry thermometer well before moving to the next test tube.
16) Repeat steps 11 – 15 in test tube #4 using a small (pea sized) piece of calcium metal.


## Waste Disposal and Lab Clean – Up:

1) Place the contents of each test tube into the properly labeled waste containers.


## The Lab Write – Up:

1) Determine all values in your data table and discuss relevant data in your conclusion.
2) For each reaction, discuss the following information in the conclusion:
      a.  Enthalpy change ($\Delta H^o$) and whether the proposed reaction was endothermic or exothermic.
      b.  Entropy change ($\Delta S^o$) and whether the proposed reaction was becoming more ordered or disordered.
      c.  Gibbs free energy change ($\Delta G^o$) and whether the proposed reaction would be spontaneous or nonspontaneous at $25^o$C.
      d.  The observations (including pH and/or temperature readings) before, during, and after the reaction to support $\Delta H^o$, $\Delta S^o$, $\Delta G^o$, and the prediction as to the spontaneity of the reaction.
3) Discuss all sources of error during this experiment and how these errors directly affected the data.

# Pre – Lab Information:

## Table #1: Standard Enthalpy and Entropy Data

| Substance (State) | Enthalpy, $\Delta H_f^o$ (kJ/mol) | Entropy, $S^o$ (J/mol●K) |
|---|---|---|
| Ca(*s*) | | |
| Ca$^{2+}$(*aq*) | | |
| Cu(*s*) | | |
| Cu$^{2+}$(*aq*) | | |
| Cl$^-$(*aq*) | | |
| H$^+$(*aq*) | | |
| H$_2$(*g*) | | |
| H$_2$O(*l*) | | |
| Mg(*s*) | | |
| Mg$^{2+}$(*aq*) | | |
| Mg(OH)$_2$(*s*) | | |
| OH$^-$(*aq*) | | |

Reference(s): _____

_____

# Sample Data Tables:

## Reaction #1: Cu($s$) + 2 H$^+$($aq$) → Cu$^{2+}$($aq$) + H$_2$($g$)

| Data Point to be Recorded/Calculated | Data Point |
|---|---|
| $\Delta H^o$ (kJ/mol) | |
| $\Delta S^o$ (J/mol•K) | |
| $\Delta G^o$ (kJ/mol) | |
| Spontaneous or Nonspontaneous? | |
| Temperature of liquid before addition of metal in degrees Celsius: | |
| Observations collected before reaction of metal: | |
| Observations collected during reaction of metal: | |
| Final temperature of liquid after addition of metal in degrees Celsius: | |

## Reaction #2: Mg($s$) + 2 H$^+$($aq$) → Mg$^{2+}$($aq$) + H$_2$($g$)

| Data Point to be Recorded/Calculated | Data Point |
|---|---|
| $\Delta H^o$ (kJ/mol) | |
| $\Delta S^o$ (J/mol•K) | |
| $\Delta G^o$ (kJ/mol) | |
| Spontaneous or Nonspontaneous? | |
| Temperature of liquid before addition of metal in degrees Celsius: | |
| Observations collected before reaction of metal: | |
| Observations collected during reaction of metal: | |
| Final temperature of liquid after addition of metal in degrees Celsius: | |

# Sample Data Tables (Continued):

## Reaction #3: Mg(s) + 2 H₂O(l) → Mg(OH)₂(s) + H₂(g)

| Data Point to be Recorded/Calculated | Data Point |
|---|---|
| $\Delta H^o$ (kJ/mol) | |
| $\Delta S^o$ (J/mol●K) | |
| $\Delta G^o$ (kJ/mol) | |
| Spontaneous or Nonspontaneous? | |
| Temperature of liquid before addition of metal in degrees Celsius: | |
| Initial pH of liquid: | |
| Observations collected before reaction of metal: | |
| Observations collected during reaction of metal: | |
| Final temperature of liquid after addition of metal in degrees Celsius: | |
| pH of resultant solution: | |

## Reaction #4: Ca(s) + 2 H₂O(l) → Ca²⁺(aq) + 2 OH⁻(aq) + H₂(g)

| Data Point to be Recorded/Calculated | Data Point |
|---|---|
| $\Delta H^o$ (kJ/mol) | |
| $\Delta S^o$ (J/mol●K) | |
| $\Delta G^o$ (kJ/mol) | |
| Spontaneous or Nonspontaneous? | |
| Temperature of liquid before addition of metal in degrees Celsius: | |
| Initial pH of liquid: | |
| Observations collected before reaction of metal: | |
| Observations collected during reaction of metal: | |
| Final temperature of liquid after addition of metal in degrees Celsius: | |
| pH of resultant solution: | |

# Experiment #12A:
# Aqueous Electrolysis

## Introduction to Electrolysis:

1) Electrolysis is the production of a chemical reaction or chemical change through the use of an electrolytic cell.
   a. Commonly, a non–spontaneous process can be converted into a spontaneous one by the introduction of an electrical current.
2) Wires, originating from a battery, are connected to two electrodes and immersed into the material to complete an electrochemical cell.
   a. The cathode is the electrode where reduction occurs.
   b. The anode is the electrode where oxidation occurs.
3) Electrolysis of a molten salt, such as sodium chloride, is a relatively simple process by which pure sodium and chlorine elements are produced from the salt.
   a. A Downs cell is the commercially implemented cell.
   b. The sodium ion, $Na^+(l)$, is reduced to pure sodium metal, $Na(l)$, at the cathode.
   c. The chloride ion, $Cl^-(l)$, oxidized to pure diatomic chlorine, $Cl_2(g)$ at the anode.
4) Unlike in the electrolysis of a molten salt, water can become involved in the electrolysis of an aqueous solution.

## Aqueous Electrolysis:

1) To determine what are the predominant oxidation and reduction processes in aqueous electrolysis, all possible half – reactions must be considered.
   a. At the cathode, the larger the reduction potential (more positive or less negative), the more easily reduced.
   b. At the anode, the larger the oxidation potential (more positive or less negative), the more easily oxidized.
2) In the electrolysis of sodium chloride solutions, the possible reactions involve $Na^+(aq)$, $Cl^-(aq)$, and $H_2O(l)$.
   a. At the cathode, the two potential half – reactions are as follows:
      i. $Na^+(aq) + e^- \rightleftharpoons Na(s)$; $E^o = -2.71$ V
      ii. $2H_2O(l) + 2e^- \rightleftharpoons H_2(g) + 2OH^-(aq)$; $E^o = -0.83$ V
      iii. Under standard conditions, the water would be reduced over the sodium ion because of a larger (more positive) reduction potential.
   b. At the anode, the two potential half – reactions are as follows:
      i. $2Cl^-(aq) \rightleftharpoons Cl_2(g) + 2e^-$; $E^o = -1.36$ V
      ii. $2H_2O(l) \rightleftharpoons O_2(g) + 4H^+(aq) + 4e^-$; $E^o = -1.23$ V
      iii. Under standard conditions, it would appear that the water would be oxidized over the chloride ion because of a larger (more positive) oxidation potential.
      iv. Because the potentials are close, increasing the concentration of NaCl and creating an overvoltage could favor the oxidation of the chloride ion.

c. At more concentrated solutions of NaCl, the following half – reactions are predominant:
   i.   $2H_2O(l) + 2e^- \rightleftharpoons H_2(g) + 2OH^-(aq)$; $E^o = -0.83$ V
   ii.  $2Cl^-(aq) \rightleftharpoons Cl_2(g) + 2e^-$; $E^o = -1.36$ V
d. The combination of the half reactions creates the following net ionic equation:
   i.   $2H_2O(l) + 2Cl^-(aq) \rightleftharpoons H_2(g) + 2OH^-(aq) + Cl_2(g)$
   ii.  $E_{cell} = -0.83$ V $+ (-1.36$ V$) = -2.19$ V

### Standard Electrode (Reduction) Potentials in Aqueous Solution at 25°C

| Reduction Half–Reaction | Standard Reduction Potential, $E^o$ (V) |
|---|---|
| $Li^+(aq) + e^- \rightleftharpoons Li(s)$ | −3.04 |
| $K^+(aq) + e^- \rightleftharpoons K(s)$ | −2.92 |
| $Ca^{2+}(aq) + 2e^- \rightleftharpoons Ca(s)$ | −2.76 |
| $Na^+(aq) + e^- \rightleftharpoons Na(s)$ | −2.71 |
| $2H_2O(l) + 2e^- \rightleftharpoons H_2(g) + 2OH^-(aq)$ | −0.83 |
| $2H^+(aq) + 2e^- \rightleftharpoons H_2(g)$ | 0.00 |
| $Cu^{2+}(aq) + e^- \rightleftharpoons Cu^+(aq)$ | 0.16 |
| $ClO_4^-(aq) + H_2O(l) + 2e^- \rightleftharpoons ClO_3^-(aq) + 2OH^-(aq)$ | 0.17 |
| $Cu^{2+}(aq) + 2e^- \rightleftharpoons Cu(s)$ | 0.34 |
| $I_2(s) + 2e^- \rightleftharpoons 2I^-(aq)$ | 0.54 |
| $Ag^+(aq) + e^- \rightleftharpoons Ag(s)$ | 0.80 |
| $Br_2(l) + 2e^- \rightleftharpoons 2Br^-(aq)$ | 1.07 |
| $O_2(g) + 4H^+(aq) + 4e^- \rightleftharpoons 2H_2O(l)$ | 1.23 |
| $Cl_2(g) + 2e^- \rightleftharpoons 2Cl^-(aq)$ | 1.36 |
| $H_2O_2(aq) + 2H^+(aq) + 2e^- \rightleftharpoons 2H_2O(l)$ | 1.78 |
| $S_2O_8^{2-}(aq) + 2e^- \rightleftharpoons 2SO_4^{2-}(aq)$ | 2.01 |

### The Aqueous Solutions:
1) 1.000 $M$ NaBr
2) 1.000 $M$ KI
3) 1.000 $M$ CuSO$_4$

### Pre–Lab Information:
1) Calculate the mass necessary to produce 100.0 mL of a 1.000 $M$ aqueous solution of sodium bromide, potassium iodide, and copper (II) sulfate.

**Procedure for the Electrolysis of Three Aqueous Solutions**:

1) Calculate the mass necessary to produce 100.0 mL of a 1.000 *M* aqueous solution of sodium bromide, potassium iodide, and copper (II) sulfate.
2) Based on the masses determined in the pre – lab, measure out the calculated quantities of each on a piece of weigh paper (or weigh boat) with an analytical balance and carefully transfer into a 100.0 mL volumetric flask.
   a. Record exact mass on the data table.
3) Add up to the 100.0 mL mark with deionized water.
4) Swirl to dissolve the salt completely. If necessary, carefully add a magnetic stir bar to the flask and place onto a hotplate to stir until dissolved.
   a. Do not allow the water to splash out.
5) Separately, transfer each solution into a labeled 150 mL beaker.
   a. Using a grease/wax pencil, label each beaker.
6) Test the pH of each solution.
   a. This can be performed by placing a glass stir rod into solution and placing a drop onto pH paper.
   b. An alternate procedure using a pH meter/probe can be implemented.
7) Clamp two alligator clips with electrical leads to each clasp of a 9 volt battery.
8) To the other end of the alligator clips, attach two graphite electrodes.
9) Separately, immerse both electrodes into each of aqueous solutions. Allow process to occur for at least 3 minutes.
   a. Do not allow both electrodes to come into contact.
   b. Record observations from both electrodes for each aqueous solution.
      i. Indicate colors, states of matter, etc.
   c. Ensure electrodes are cleaned thoroughly between each electrolysis.
10) After electrolysis, remove electrodes and test pH of the resultant solution.
    a. This can be performed by placing a glass stir rod into solution and placing a drop onto pH paper.
    b. An alternate procedure using the laptop computers with pH probes can be implemented.
11) For each electrolysis, include the following:
    a. All possible half – reactions at the cathode
       i. Considering the electrode potentials and reaction conditions, select the predominant half – reaction and provide rationale.
    b. All possible half – reactions at the anode
       i. Considering the electrode potentials and reaction conditions, select the predominant half – reaction and provide rationale.
    c. The net ionic equation (for each electrode)
    d. Combined balanced half reactions with states of matter

**Waste Disposal and Lab Clean – Up**:

1) Place all solutions into the properly labeled waste containers.
2) All electrodes should be carefully rinsed with water and dried.
3) The electrode with electroplated copper should be placed into a beaker so that nitric acid can be applied to remove.

**The Lab Write – Up**:

1) Determine all values in your data table and discuss relevant data in your conclusion.
2) For each electrolysis, include the following and discuss in the conclusion:
   a. All possible half–reactions at the cathode
      i. Considering the electrode potentials, select the predominant half – reaction and provide rationale.
   b. All possible half – reactions at the anode
      i. Considering the electrode potentials, select the predominant half – reaction and provide rationale.
   c. The net ionic equation (for each electrode)
   d. Combined balanced half reactions with states of matter
3) In the electrolysis of KI and NaBr, pure elemental iodine, potassium, and sodium cannot be isolated. Discuss by using the appropriate balanced equations when applicable.
4) Discuss the pH changes for each solution. Reflecting back on pH of aqueous salts, do the initial pH measurements for each aqueous solution make sense? Explain.
5) Discuss the electrolysis of calcium perchlorate. Indicate the half–reactions that occur at each electrode. Determine the combined balanced half reaction and calculate $E_{cell}$.
   a. Assume a 1.0 *M* concentration of calcium perchlorate and an overvoltage.
6) Discuss all sources of error during this experiment and how these errors directly affected the data.

## Pre – Lab Information:

| Data Point | NaBr Solution | KI Solution | CuSO$_4$ Solution |
|---|---|---|---|
| Calculated mass of salt to produce 100.0 mL of a 1.000 *M* solution in grams: | | | |

## Sample Data Table:

| Data Point | NaBr Solution | KI Solution | CuSO$_4$ Solution |
|---|---|---|---|
| Mass of salt measured via an analytical balance in grams: | | | |
| Molarity of salt solution in moles per liter: | | | |
| Initial pH (before electrolysis): | | | |
| Observations of cathode: | | | |
| Observations of anode: | | | |
| pH of resultant solution (after electrolysis): | | | |
| Possible reduction reactions with E$^o$ | | | |
| Possible oxidation reactions with E$^o$ | | | |

# Net Ionic Equations and $E_{cell}$ for Data:

| Solution | Combined Balanced Half Reactions | $E_{cell}$ (Volts) |
|---|---|---|
| NaBr Solution | | |
| KI Solution | | |
| CuSO$_4$ Solution | | |

# Net Ionic Equation & $E_{cell}$ for Post–Lab Question:

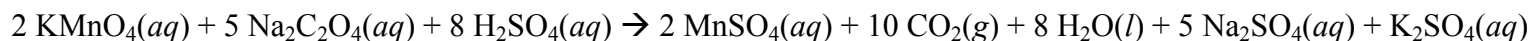| Solution | Combined Balanced Half Reactions | $E_{cell}$ (Volts) |
|---|---|---|
| calcium perchlorate solution | | |

# Experiment #12B:
# An Oxidation – Reduction (Redox) Titration;
# The Determination of an Unknown Oxalate Concentration

**Introduction to Redox**:
1) Oxidation–reduction (redox) reactions are those that involve electron transfer between species or atoms that result in a change in oxidation numbers.
    a. **Oxidation** is the process where there is a loss of electrons by a species (or an increase in the oxidation number of an atom.)
    b. **Reduction** is the process where there is a gain of electrons by a species (or a decrease in the oxidation number of an atom.)
2) In a redox reaction, the loss of electrons from one species results in the gain of electrons from another species.
    a. An **oxidizing agent** is the group that oxidizes another species and thus becomes reduced.
    b. A **reducing agent** is a group that reduces another species and thus becomes oxidized.
3) The oxidation number (or oxidation state) is defined as the actual charge of the atom if it exists as a monoatomic ion or a hypothetical charge assigned to the atom in a substance.
4) To assign the oxidation numbers, a set of rules have been devised.
    a. Elements – Any atom in its elemental form has an oxidation number of zero.
    b. Monatomic ions – oxidation numbers are equal to the charge of the ions.
    c. Oxygen – for most compounds, the oxidation number is 2–.
        i. In some instances, such as $H_2O_2$, it is 1–.
    d. Hydrogen – for most compounds, the oxidation number is 1+.
        i. When bound to metals, however, the oxidation number is 1–.
    e. Halogens – for most binary compounds, they have an oxidation number of 1–.
        i. Only when Cl, Br, and I are the central atom bound to other halogens above them in the periodic chart do they differ from this scheme.

## The Balanced Equation:

$$2\ KMnO_4(aq) + 5\ Na_2C_2O_4(aq) + 8\ H_2SO_4(aq) \rightarrow 2\ MnSO_4(aq) + 10\ CO_2(g) + 8\ H_2O(l) + 5\ Na_2SO_4(aq) + K_2SO_4(aq)$$

1) Potassium permanganate ($KMnO_4$) reacts with sodium oxalate in the presence of a sulfuric acid to produce carbon dioxide, water, and aqueous salts.

## Pre – Lab Information:
1) From the balanced equation, determine the chemical/ion undergoing oxidation, chemical/ion undergoing reduction, chemical/ion defined as the oxidizing agent, and the chemical/ion defined as the reducing agent.
2) Provide the net ionic equation (balanced redox reaction) from the balanced equation above.
3) Calculate the mass of potassium permanganate necessary to prepare 500.0 mL of a 0.02000 *M* aqueous solution.

## Part 1–Preparation of a 0.02000 *M* Solution of $KMnO_4$:

1) Using the calculated mass from the pre-lab, measure out this mass of $KMnO_4$ on a piece of weigh paper (or weigh boat) with an analytical balance.
    a. Record exact mass in the lab notebook.
    b. Never add solute to the weigh paper INSIDE of the balance; do this outside the balance and then place onto the balance.
2) Carefully transfer powder into a 500.0 mL volumetric flask.
3) Add up to the 500.0 mL mark with deionized water.
4) Swirl to ensure potassium permanganate is completely dissolved.

## Part 2–Procedure for Redox Titration of an Oxalate Solution of Unknown Concentration:

1) Pipet exactly 25.0 mL of an unknown oxalate solution into a 400 mL beaker.
2) To the same 400 mL beaker, add about 75.0 mL of deionized water followed by 25.0 mL of a 3.0 *M* $H_2SO_4$ solution which has been "spiked" with a catalytic amount (0.030 *M*) of $MnSO_4$.
3) Insert a magnetic stir bar into the solution and place onto a stirring hotplate. Turn on stir function and heat.
4) Heat the solution to maintain a temperature of around $80^oC$.
    a. During the titration, the oxalate solution can remain on the hotplate but ensure that the solution does not boil.
5) While the oxalate solution is heating, place an empty 100 mL beaker under a buret attached to a buret stand.
6) Ensure the buret stopcock is in the closed position. Using a short stem funnel, add approximately 10.0 mL of the prepared $KMnO_4$ solution to the buret.
7) Condition the buret by holding it horizontally and rotating to allow the $KMnO_4$ solution to wet the inside of the glass completely. Discard the rinse into a properly labeled "$KMnO_4$ waste" container.
8) Fill the buret with the prepared $KMnO_4$ solution and adjust the meniscus to zero.
    a. Record the initial buret reading as 0.00 mL.
    b. Keep any remaining $KMnO_4$ solution for later.
9) Once the oxalate solution has achieved a stable temperature of $\sim 80^oC$, move the buret into place over top of the oxalate solution.
10) Add 1.0 mL allotments of the $KMnO_4$ solution and allow pink or purple color to disappear.
11) Titrate until the endpoint has been reached.
    a. The endpoint of the titration is when the addition of a single added drop of $KMnO_4$ solution produces a faint but permanent pink or purple color.
12) Record the total volume of $KMnO_4$ added.
13) Repeat titration for a second trial.
    a. If results differ significantly from the first two trials or if time permits, repeat for a third trial.
14) Determine the oxalate concentration of the unknown solution.

## Waste Disposal and Lab Clean – Up:
1) Place all solutions together into the properly labeled waste container.


## The Lab Write – Up:
1) Determine all values in your data table and discuss relevant data in your conclusion.
2) For the first part of the experiment:
   a. Calculate the molarity of the prepared $KMnO_4$ solution. How close was it to the desired molarity of 0.02000 *M*?
3) For the second part of the experiment:
   a. Determine the molarity of the oxalate anion in the unknown solution.
4) Determine the the chemical/ion undergoing oxidation, chemical/ion undergoing reduction, chemical/ion defined as the oxidizing agent, and the chemical/ion defined as the reducing agent. Discuss this information in your conclusion.
4) Provide the net ionic equation (balanced redox reaction) from the balanced equation above.
5) Discuss the entropy, enthalpy, and the Gibb's free energy of this reaction using the following equation. Predict the sign on each.
   a. $\Delta G = \Delta H - T\Delta S$
6) Discuss all sources of error during this experiment and how these errors directly affected the data.

# Data Table for Pre – Lab:

| Redox Label | Chemical/Ion Responsible |
|---|---|
| Chemical/Ion undergoing oxidation: | |
| Chemical/Ion undergoing reduction: | |
| Chemical/Ion defined as the oxidizing agent: | |
| Chemical/Ion defined as the reducing agent: | |

Net Ionic Equation (Balanced Redox Reaction):

_____

Mass of potassium permanganate ($KMnO_4$) necessary
to prepare 500.0 mL of a 0.02000 $M$ aqueous solution in grams:

_____

# Sample Data Tables:

## Part 1–Preparation of a 0.02000 *M* Solution of KMnO₄:

| Data Point to be Recorded/Calculated | Data Point |
|---|---|
| a) Mass of potassium permanganate ($KMnO_4$) measured in grams: | |
| b) Moles of potassium permanganate ($KMnO_4$) measured in mol: | |
| | |
| c) Total volume in volumetric flask in liters: | |
| | |
| d) Molarity of potassium permanganate ($KMnO_4$) solution prepared in moles per liter (b/c): | |

## Part 2–Procedure for Redox Titration of an Oxalate Solution of Unknown Concentration:

| Data Recorded/Calculated | Trial #1 | Trial #2 | Trial #3 |
|---|---|---|---|
| a) Molarity of potassium permanganate ($KMnO_4$) measured in moles per liter:  (See d) in part 1) | | | |
| b) Volume of unknown oxalate solution titrated in milliliters: | 25.00 mL | | |
| | | | |
| c) Volume of potassium permanganate ($KMnO_4$) solution added in titration of oxalate solution in milliliters: | | | |
| d) Moles of potassium permanganate ($KMnO_4$) added in titration of oxalate solution in mol[*]: | | | |
| | | | |
| e) Moles of sodium oxalate ($Na_2C_2O_4$) titrated by potassium permanganate ($KMnO_4$) in mol[†]: | | | |
| f) Molarity of sodium oxalate ($Na_2C_2O_4$) titrated by  potassium permanganate ($KMnO_4$) in moles per liter: | | | |
| g) Average molarity of sodium oxalate ($Na_2C_2O_4$) titrated by  potassium permanganate ($KMnO_4$) in moles per liter: | | | |

[*]Calculated by using calculated molarity from part 1
[†]Calculated by using the mole to mole ratio in balanced equation

# Experiment #13:
# The Synthesis and Characterization of Aspirin
# (Acetylsalicylic Acid)

## An Introduction to Aspirin (Acetylsalicylic Acid):

1) Aspirin (acetylsalicylic acid) is one of the oldest and most versatile drugs known to medicine.
2) Aspirin (acetylsalicylic acid) is an analgesic, antipyretic, anti–inflammatory drug.
3) It is also used as an antiplatelet agent as it inhibits platelet aggregation.
4) It works by preventing the synthesis of prostaglandins by inhibiting both the cyclooxygenase–1 (COX–1) and cyclooxygenase–2 (COX–2) isoenzymes
   a. COX–2 mediates tissue inflammation.
   b. COX–1 mediates platelet aggregation, gastric protection, and induction of renal function.
5) Aspirin is in a class of medications called salicylates.
   a. Common adverse effects include: gastrointestinal (GI) distress (including upset stomach, heartburn, nausea, vomiting).
   b. Less common severe adverse effects include: tinnitus, Reye syndrome, GI ulceration, allergic reactions (including hives, shortness of breath, swelling, and/or anaphylaxis.)
6) Aspirin should not be taken with other salicylates, non–steroidal anti–inflammatory drugs (NSAIDs) such as ibuprofen or naproxen sodium, or anticoagulants such as Coumadin (warfarin).
7) Aspirin can be purchased both over–the–counter (OTC) and by prescription (Rx) from a physician.
   a. OTC preparations: Aspergum, Bayer Aspirin, Bufferin, Ecotrin, Empirin, Halfprin, St. Joseph Aspirin, and many generics.
   b. Rx preparation: ZORprin
8) Aspirin is synthesized from the acetylation (using acetic anhydride or acetyl chloride) of salicylic acid.
   a. Salicylic acid is a natural product that can be derived from the bark of a willow tree.
   b. Salicylic acid is not used as an analgesic due to the highly acidic nature as it is damaging to mucous membranes.
      i. This is why this chemical is used as a ketatolytic (peeling) agent in the treatment of acne, corns, and warts.
   c. The addition of the acetyl group to the phenolic oxygen makes the medication less irritating to the esophagus and gastric lining.
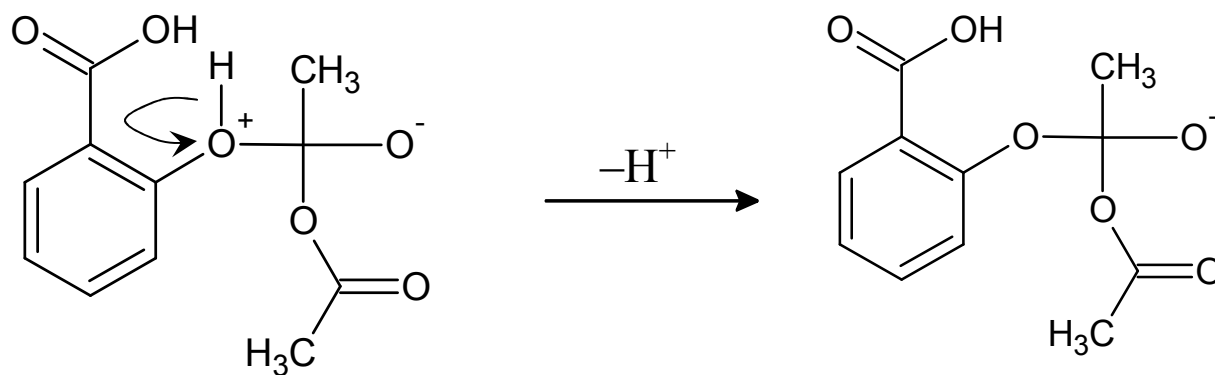
# The Balanced Equation:



**Salicylic Acid**  +  **Acetic Anhydride**  $\xrightarrow{H_3PO_4}$  **Acetylsalicylic Acid**  +  **Acetic Acid**
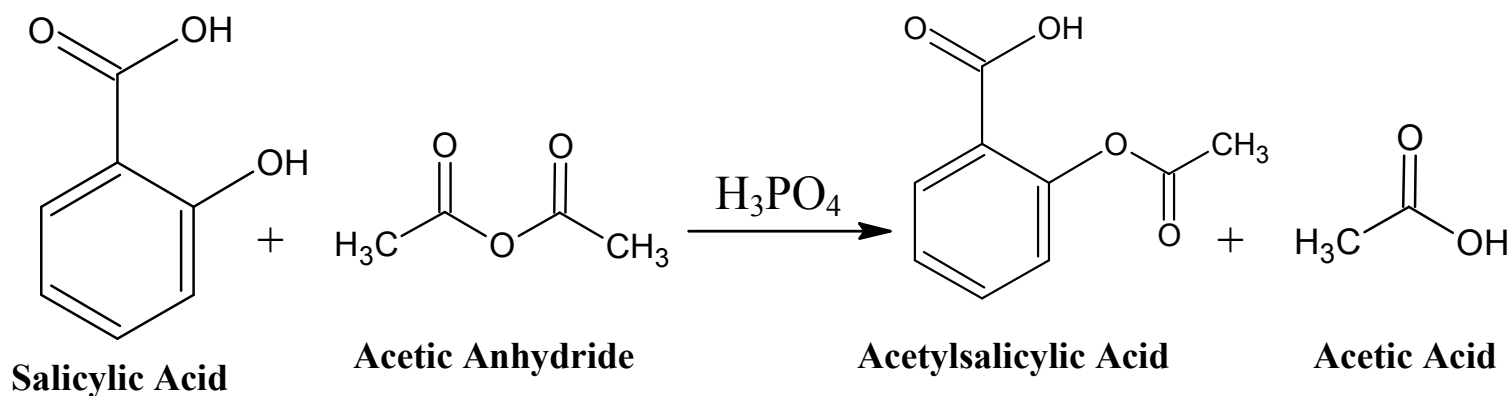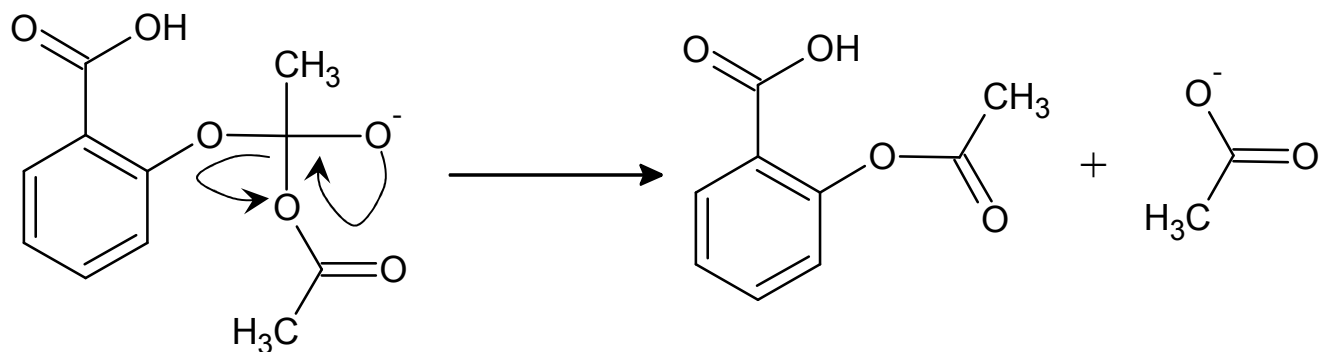
# The Reaction Mechanism:
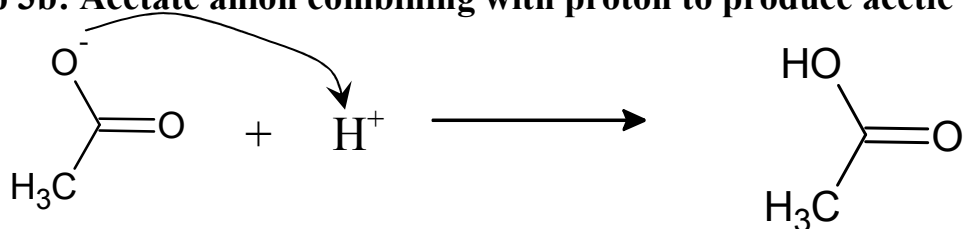**Step 1: Attack of phenolic oxygen on carbonyl of acetic anhydride**
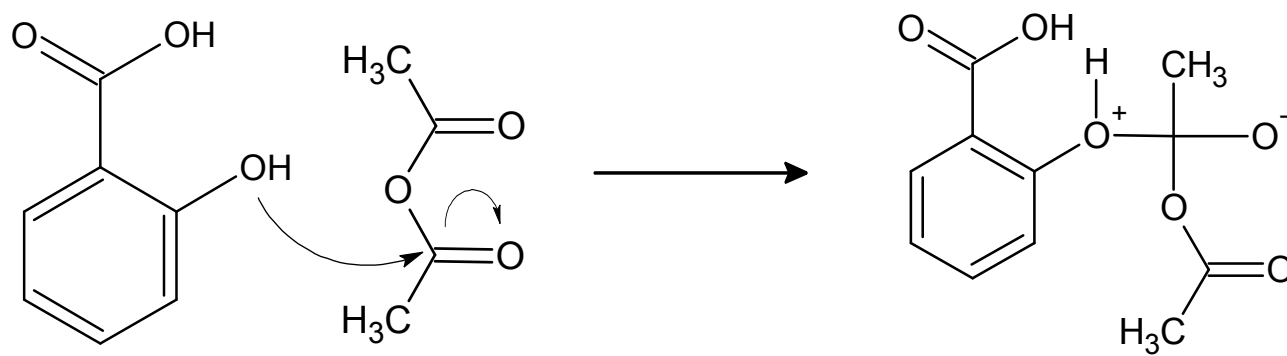


**Step 2: Loss of phenolic proton**



$-H^+$

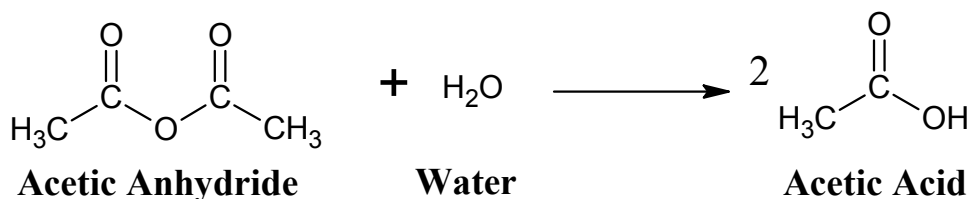## Step 3a: Break down of tetrahedral intermediate and ejection of acetate leaving group



## Step 3b: Acetate anion combining with proton to produce acetic acid



# The Side Reaction:

1) Hydrolysis of acetic anhydride can occur and the introduction of water should be prematurely avoided.



**Acetic Anhydride**      **Water**      **Acetic Acid**

# The Experimental Procedure for the Synthesis of Aspirin:

1) Create a hot water bath with a 400–600 mL beaker on a hot plate.
   a. Maintain the temperature between 70–80°C and monitor temperature with an alcohol thermometer.
2) Measure out 4.00 g of salicylic acid on a balance and transfer into a 125 mL Erlenmeyer flask.
3) Using a 10.0 mL graduated cylinder, measure out 8.0 mL of acetic anhydride and pour into the flask containing the salicylic acid.
4) Next, place 10 drops of concentrated phosphoric acid into the flask and swirl gently.
5) Clamp the flask onto a ring stand or the metal scaffolding and immerse into the hot water bath.
   a. Do not allow water to get into the flask!

6) Keep immersed in the hot water bath for 20 minutes and using a glass stirring rod, stir the solution periodically.
7) After 20 minutes, carefully add 5.0 mL of deionized water to the hot solution to decompose any excess acetic anhydride if present.
8) After five minutes, remove from the hot water bath and allow to cool on the lab bench.
9) During this cooling step, obtain 50.0 mL of deionized water.
10) In addition, create an ice–water bath with a 400–600 mL beaker.
11) After the flask has cooled for a few minutes, pour the 50.0 mL of water into the flask to hasten crystallization.
12) Next, clamp the flask into the ice–water bath and swirl occasionally.
13) Allow at least five minutes to pass to permit maximal aspirin crystal formation.
14) Chill approximately 25.0 mL of deionized water in another ice–water bath.
15) While your crystals are forming, obtain a piece of filter paper and pre–weigh the filter paper on a watch glass.
    a. Record this value in your data table.
16) Place the filter paper into a Büchner funnel and set up a vacuum filtration system.
    a. See attached diagram.
17) Turn on the vacuum and then wet the filter with a small amount of chilled deionized water.
18) Slowly transfer the contents of the flask onto the filter paper.
    a. The liquid should pass through the filter paper leaving the aspirin behind.
19) After the flask is empty, use the cold 25.0 mL deionized water to rinse out the flask and place the contents into the Büchner funnel.
20) Once all of the water has passed through the filter and the crystals look reasonably dry, remove the vacuum and transfer the aspirin and filter paper onto the pre–weighed watch glass.
21) Allow the aspirin to dry in an oven as directed by your professor.
22) Once the sample is dry, run melting point determination to characterize and assess purity.

## The Experimental Procedure for the Purity Assessment of Aspirin:

1) Run a melting point on the aspirin when it is completely dry.
    a. Using a capillary tube (with one open and one closed end), invert the open end into powder sample and press downward.
    b. Once the required amount of powder is in the tube, invert and tap the powder down into the closed end.
    c. The column of powder in capillary tube should be 2–3 mm, tightly packed.
2) Place packed capillary tube into the melting–point apparatus and turn on the heating dial.
3) The rate of heating is important; heat no faster than $1.0^{\circ}C$ per minute.
    a. If the approximate melting point is known, the temperature can be increased more rapidly at the beginning until the temperature is between $10.0–20.0^{\circ}C$ below the anticipated temperature. At this point, the rate of heating should be slowed.
    b. Most melting points, even for pure substances, are reported as a range.
4) The melting point begins when the first drop of liquid is detected. The end point is when the entire solid sample becomes a liquid.
5) Record the melting point range of aspirin in your data table.
6) Determine the limiting reactant and calculate the theoretical yield and percent yield.

## Waste Disposal and Lab Clean – Up:

1) Combine all aqueous filtrates, dilute with plenty of water, and flush down the drain with plenty of water.
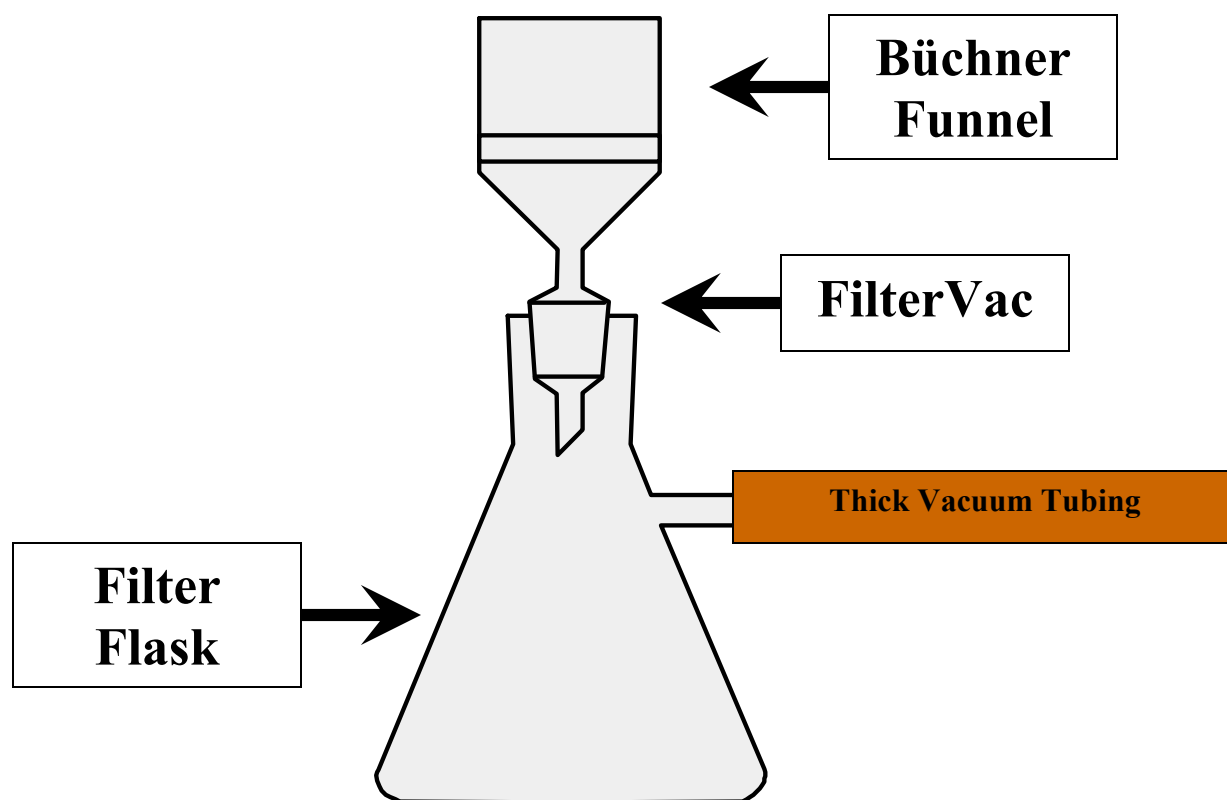2) The aspirin product should be placed into the appropriately labeled waste container.


## The Lab Write – Up:

1) Determine the theoretical yield, actual yield, and percent yield, place all values into a data table, and discuss all relevant data points in your conclusion.
    a. Discuss potential places for error that accounted for low percent yield.
    b. Percent yield = (Actual Yield/Theoretical Yield) * 100%
2) Determine the melting point of product, the true value melting point, and place into a data table. In your conclusion, discuss this information and how this relates to the purity of your product.
    a. Calculate the percent error with the following formula: Percent error = [(true value – experimental value)/true value] * 100%
    b. Discuss potential places for error that decreased the purity of the product.
3) How would you account for the smell of vinegar upon opening a bottle of expired aspirin?

# Sample Data Table:

| Data Point to be Recorded/Calculated | Data Point |
|---|---|
| a) Mass of salicylic acid in grams: | |
| b) Moles of salicylic acid in mol: | |
| | |
| c) Volume of acetic anhydride in milliliters: | |
| d) Density of acetic anhydride in grams per milliliters: | |
| e) Mass of acetic anhydride in grams: | |
| f) Moles of acetic anhydride in mol: | |
| | |
| g) Drops of phosphoric acid: | |
| | |
| h) Mass of filter paper + watch glass in grams: | |
| i) Mass of filter paper + watch glass + aspirin product after drying in grams: | |
| j) Mass of aspirin (actual yield) in grams: | |
| | |
| k) Limiting reactant: | |
| *l*) Theoretical yield of aspirin in grams: | |
| m) Percent yield of aspirin (j/*l* x 100%) in %: | |
| | |
| n) Experimental melting point range of aspirin in degrees Celsius: | |
| o) True melting point point/range of aspirin in degrees Celsius: | |
| p) Percent error range for melting point of aspirin in %: | |

# Model of Vacuum Filtration Using a Büchner Funnel

**Büchner Funnel**

**FilterVac**

**Thick Vacuum Tubing**

**Filter Flask**

Clamp filter flask securely to a ring stand before affixing vacuum tubing and turning on vacuum.