General Chemistry II
Laboratory Manual

Winter term 2011-12
Lab begins the *first* week of classes

Required Text for
CHEM 132
(All sections)

You must bring this lab manual, plus safety glasses, to the first lab period.

Safety glasses are required, and they are sold at the bookstore.
Department of Chemistry & Biochemistry: Laboratory Safety Policy and Rules

1. All students will complete the safety training segment prior to starting laboratory work. You must know the location of safety equipment: fire extinguishers, safety showers, eye wash facilities, fire blanket and first aid kit.

2. Approved eye protection (safety glasses/goggles) must be worn at all times in the laboratory. Even if you have completed your lab work, eye protection must remain on in the lab.

3. Stools are not permitted in the laboratory. The only exception to this is near the melt-temp instrumentation in the Organic lab. If stools are moved, they will be moved from the lab.

4. No eating, drinking any liquids (including water), chewing gum or smoking in the laboratory.

5. No bare feet in the laboratory; open-toed sandals are dangerous and are not permitted. Long pants or skirts must be worn. If shorts are worn, you may cover your legs with a knee-length lab coat or apron. Latex gloves are also available for any student who wishes to use them.

6. Long hair must be kept tightly in place. Hair and loose clothing can catch fire easily.

7. Do not enter the lab unless the instructor or supervisor is present.

8. No unauthorized experiments are to be performed. Alterations to an existing lab must be approved by the instructor.

9. Any accident or injury must be reported to the supervisor at once.

10. Chemicals are generally to be kept in designated access areas. Caps are to be replaced promptly on any reagents used in the lab. Use care when transferring or dispersing chemicals. All spills must be cleaned up promptly and completely.

11. If any material is spilled on the skin, wash it off immediately with large volume of water. Notify your instructor.

12. Reading labels and using the exact chemical in its proper concentration is the responsibility of the student. For some reagents, only the instructor is allowed to disperse them (these will be announced in the lab introduction).

13. Do not use cracked or chipped glassware. Dispose of it in a proper manner as indicated by your instructor.

14. Proper clean up and maintaining a well-ordered work space is essential to lab safety. Bench tops and the weighing areas are to be wiped clean. Clean all shared equipment. Pay special attention to the proper cleaning techniques for various pieces of instrumentation. Improper cleaning can damage expensive equipment and render it useless!

15. Instrumentation can only be used after instructor approval or according to any instructions given in the lab introduction.

16. Proper disposal of waste chemicals is the student’s responsibility. If there is a question, ask the supervisor.

17. Read the entire experiment and complete any pre-laboratory assignments before entering the laboratory. These include, for the Organic laboratory, a truncated procedure written in pen in your lab notebook.

18. Inform the instructor immediately of any broken thermometers immediately. If a mercury thermometer is broken, any spills must be cleaned up by the instructor.

19. Any special health factors such as an allergic reaction to a chemical or a pregnancy must be reported to the instructor as soon as possible.

20. Always use common sense in the laboratory. If something is unclear, be sure to ask the instructor before proceeding.
Lab Supplement: Tips on using Excel

Several of the laboratories require you to analyze your data using the Excel software available in the Zurn Scientific Computing Lab (ZSCL). The most common type of analysis that you will be performing is called a linear regression. This analysis determines the equation of the “best-fit” straight line through your data points.

1. Open the Excel program.
2. Use the topmost cells to label the data columns, this will make it easier for you to keep track of the data. Include units with your data labels. A column can be widened by selecting the top of it and dragging with the mouse.
3. Enter your x-coordinate data into the column on the left. Do not include units in cells with numbers.
4. Enter your y-coordinate data into the column on the right.
5. If the data is formatted incorrectly (such as too many/too few decimal places), use your mouse to select the cells with data that you wish to change. You may select entire columns. Under the Number options, select Number as the category and then enter the correct number of decimal places.
6. To chart, use the mouse to select the entirety of data, do not select the labels.
7. Select Insert, and then Scatter Chart with only markers.
8. The plot of your data should now appear superimposed over your window, if it doesn't look like the correct data was plotted, try the process again. If it does, you should format your chart to appear as you would like it.
9. With the chart selected, you can select the Layout to give a chart title, label the axes, format the axes, change the background and remove/add gridlines.
10. With the chart selected, you can select Design to change the data presented and label the data series in the legend. (To do this, choose Select Data, then select your data and Edit it to provide a name).
11. To perform a linear regression, go to the Layout area, and select Trendline under analysis. Move down to “More Trendline Options” and select “Display Equation on Chart” and the Linear Trend.
12. The equation of your line should now appear on your graph. You can move it to where it is read most easily.
13. To remove a chart that is incorrect, click on the chart, then select, Edit, Clear, All.
14. When you are done, check with your instructor about either printing or e-mailing your chart and data.

Using Equations
1. To use equations in a cell, select the cell within which you want a value to be calculated.
2. Type the formula using other cell labels, preceded by an "=" sign.
3. For more complicated formulas, use parentheses.

EXAMPLES
a) Suppose I wanted to add together the contents of cell A1 and B3. Type into the cell where the result should be:
   =A1+B3
b) Suppose I wanted to multiply A1 by B1 and then subtract away the contents of B2. Type:
   =(A1*B1) – B2
c) Suppose I want to take the average of cells A1 to A5, then divide the result by 3.47. Type:
   =(AVERAGE(A1:A5)) / 3.47
**EXPERIMENT 10 – Using Stoichiometry to Predict the Products of Reaction**

**Goal** To use gravimetric methods to determine the identity of a solid product.

**Objectives** After completion of this experiment, students will be able to do the following:

1. Use stoichiometry concepts to determine which of several possible reactions occurred, based on the mass of the product obtained.

2. Review stoichiometry and limiting reagent calculations.

3. Define: (a) hydrate; (b) monohydrate; (c) dihydrate; (d) anhydrous; (e) crystalline solid.

4. Given the mass of a sample of a hydrate and the mass of the anhydrous form of the hydrate, calculate the formula of the hydrate compound.

**INTRODUCTION**

Stoichiometry can often be a useful tool in predicting the products of a reaction. In this two-part experiment, you will perform two reactions that will be analyzed using the reaction stoichiometry. In Part I, you will isolate an insoluble product using the process of gravity filtration. In Part II, you will decompose a hydrated form of a solid by heating it. In both parts, you will need to take careful measurements of the mass of reactants and products for analysis. You will then use stoichiometry to make conclusions concerning the reactions that you have observed. You will be responsible for designing and performing the appropriate calculations in order to reach your conclusions.

You will be graded in part upon your quantitative results in this lab, so it is important that you are as careful and complete as possible.

**Part I: The Identity of an Insoluble Precipitate**

One easily seen signal of a chemical reaction is the formation of an insoluble precipitate. This experiment deals with a quantitative interpretation of a reaction in which this signal has appeared. In this experiment, you will examine the reaction between Ba(NO$_3$)$_2$ and NH$_2$SO$_3$H (sulfamic acid) in a hot solution. The identity of the insoluble substance that results from the reaction will be determined from mass relationships.

Known quantities of Ba(NO$_3$)$_2$ and NH$_2$SO$_3$H will be allowed to react in boiling water. Barium nitrate is an ionic compound and sulfamic acid is a covalent compound. Certain covalent bonds in the molecules of sulfamic acid will break slowly during this reaction, and a polyatomic anion will be formed. This anion will combine with the Ba$^{2+}$ cations from Ba(NO$_3$)$_2$ to form an ionic substance that appears as a white precipitate.

All of this precipitate must be separated slowly by gravity filtration because its mass must be measured. Since you will need to know the mass of the precipitate to determine its identity, you must remove all of the residual water by drying. You will know when the precipitate is dry because its mass will reach a constant value within ± 0.05 g.
The precipitate will be formed from one of three possible reactions:

a. \( \text{_____ Ba(NO}_3\text{)}_2(aq) + \text{_____ NH}_2\text{SO}_3\text{H(aq)} \rightarrow \text{_____ Ba(NH}_2\text{SO}_3\text{)}_2(s) + \text{_____ HNO}_3(aq) \)

b. \( \text{_____ Ba(NO}_3\text{)}_2(aq) + \text{_____ NH}_2\text{SO}_3\text{H(aq)} + \text{_____ H}_2\text{O(l)} \rightarrow \) 
\( \text{_____ BaSO}_4(s) + \text{_____ NH}_4\text{NO}_3(aq) + \text{_____ HNO}_3(aq) \)

c. \( \text{_____ Ba(NO}_3\text{)}_2(aq) + \text{_____ NH}_2\text{SO}_3\text{H(aq)} + \text{_____ H}_2\text{O(l)} \rightarrow \) 
\( \text{_____ Ba(NH}_2\text{)}_2(s) + \text{_____ H}_2\text{SO}_4(aq) + \text{_____ HNO}_3(aq) \)

The reactions are unbalanced (you will balance them in the pre-lab). From the experimental data, you will be able to determine which one actually occurs from the masses of the precipitate and the limiting reactant.

**PROCEDURE**

**Getting Started**

1. Your laboratory instructor will tell you how you will heat your solutions, and how to set up the gravity filtration apparatus.

**Initiating the Reaction**

1. You will need about 0.65 - 0.70 g of Ba(NO\text{3})\text{2} and about 1.2 - 1.3 g of NH\text{2}SO\text{3}H. One of these substances is the limiting reactant, as you should be able to calculate. Use the top-loading digital balances in the lab (not the Mettler balances in the weighing room) for all measurements of mass. Use weighing paper in all cases. Record each mass that you measure neatly in a data table in the Data and Results page.

2. Transfer both samples to a 250-mL beaker and add 100 mL of distilled water from a graduated cylinder.

3. Stir the mixture until most of the solids have dissolved. The remainder will dissolve when the solution is heated.

4. Record the level of the solution in the beaker. You will want to maintain a constant volume of liquid throughout the course of the reaction.

5. If a magnetic stirrer/heater is available, use it to heat your reaction. If such a stirrer is not available, use a piece of wire gauze on top of a tripod or on a ring supported by a ring stand.

    **Caution:** Avoid burning your fingers. Do not touch the tripod or the wire gauze at any time while the solution is being heated. Constantly stir the solution while it is being heated.
6. Heat your solution to a gentle boil. Allow the solution to boil for 30 minutes.

7. If you are not using a magnetic stirrer, you will need to stir the solution in a nearly constant fashion with a stirring rod. Add increments of distilled water to maintain the original volume.

**Finishing the Experiment**

1. When the 30 minute heating period is completed, allow the system to cool.

2. While the beaker and its contents are cooling, obtain the mass of a piece of filter paper using a top-loading balance. You will use this filter paper to separate the precipitate from the solution. Record the mass of your filter paper in your data table. Also obtain a clean, empty 150 mL beaker.

3. Filter the cooled mixture containing the precipitate. You may want to use a rubber policeman to assist removing the precipitate from the walls of the beaker.

4. Use a spatula to loosen the edge of the filter paper from the filter funnel.

5. Carefully transfer the filter paper and its contents to the 150 mL beaker. The paper should be upright, never upside down.

6. Place the beaker in an oven at 100 °C and allow it to remain there for twelve minutes. Be sure to label your beaker so you can identify it later!

7. During this time, you can calculate the limiting reactant. You can also calculate the theoretical yields of your three possible solid products: Ba(NH₂SO₃)₂, BaSO₄, and Ba(NH₂)₂. Do this neatly in your calculations section.

8. After the twelve minutes, remove the beaker from the oven, using tongs or oven gloves. Allow the beaker to cool by setting it on wire gauze on the benchtop. Obtain and record the mass of the filter paper and its contents.

9. Place the beaker back into the oven and allow the sample to dry for an additional eight minutes. Remove the beaker from the oven and allow it to cool. Reweigh the sample. Your mass must be within ± 0.05 gram of the initially recorded mass. If it has lost more than 0.05 gram during the second heating, put the sample back into the oven, reheat for eight minutes, and reweigh your sample. Use the lightest weight for your final mass. Do not dispose of your

   **Caution:** Before you leave the laboratory, make sure that your gas outlet and those of your neighbors are closed.
**Part II: The decomposition of a hydrate**

A **hydrate** is a salt that has crystallized from aqueous solution with weakly bound water molecules contained in the crystals. The water molecules present are in a definite stoichiometric ratio with respect to the other atoms present in the compound.

Consider the crystallization of sodium acetate (NaC$_2$H$_3$O$_2$) from aqueous solution as an example. The crystals that form can be removed from the solution by filtration. If these crystals are then allowed to sit in the open air for a few hours, all of the residual moisture on their surfaces will evaporate. They will appear to be dry. Nevertheless, water will still be present, and it will be present in definitive stoichiometric amount. Chemical analysis would show that three water molecules accompany every formula unit of sodium acetate. The compound is an example of a hydrate, and its formula in the solid state is written as NaC$_2$H$_3$O$_2$·3H$_2$O(s). This compound is called sodium acetate trihydrate. The solid form of this compound with no H$_2$O present, NaC$_2$H$_3$O$_2$(s), is called the anhydrous form. The anhydrous solid can be produced by heating the hydrated solid.

The definite stoichiometry occurs because the water in a hydrate occupies definite sites in the crystalline lattice, just as Na$^+$ and Cl$^-$ ions occupy definite positions in the NaCl lattice. Since this water occupies definite sites, it must be present in a definite stoichiometric amount. The quantity of water will not change as long as the temperature (and pressure) is not altered significantly. A substantial increase in temperature, however, will cause the loss of hydrate water. You will see this phenomenon in today’s experiment.

**PROCEDURE**

1. Weigh an empty test tube (one with a lip) using an analytical balance (set the test tube in the two grooves so it doesn’t slide around).

2. Place 2-3 g of hydrated copper(II) sulfate salt into the test tube and record the exact mass added ± 0.0001 g.

3. Clamp the test tube at the top (so the lip keeps it from sliding off) with a test tube holder and carefully heat in a Bunsen burner flame for 10-15 minutes. Heat slowly at first, be sure to move the test tube around in the flame often. Do not char the sample by heating too much in any one place. Take care not to point the test tube towards yourself or your neighbors.

4. Place the test tube containing the salt into a beaker to cool. Do NOT pour the salt out of the test tube! Allow to cool for at 10-15 minutes.

5. Reweigh the test tube and salt.

6. Reheat the sample for another 10 minutes. Cool in a beaker as before and reweigh.

7. Continue this process until the two weighings differ by no more than 0.05 g. Record your final mass and any observations. Do not dispose of your product until you have completed the calculations.

8. Determine the molecular formula of the hydrated form of copper(II) sulfate. To do this, you will need to determine the number of water molecules present per each CuSO$_4$ formula unit in the reactant. You will write the molecular formula of the hydrated complex:

   \[
   \text{CuSO}_4\cdot x \text{H}_2\text{O(s)}
   \]

   where \(x\) is the number of water molecules present per CuSO$_4$ formula unit.
Experiment 10 – Pre-lab Assignment

Name: ___________________________  Lab Day and Time: _____________

1. The following reactions, shown in unbalanced equations, are pertinent to this experiment. Balance the equations. (Hint: One of them is already balanced!) (1 pt)

   a. _____ Ba(NO$_3$)$_2$(aq) + _____ NH$_2$SO$_3$H(aq) $\rightarrow$ _____ Ba(NH$_2$SO$_3$)$_2$(s) + _____ HNO$_3$(aq)

   b. _____ Ba(NO$_3$)$_2$(aq) + _____ NH$_2$SO$_3$H(aq) + _____ H$_2$O(l) $\rightarrow$

   _____ BaSO$_4$(s) + _____ NH$_4$NO$_3$(aq) + _____ HNO$_3$(aq)

   c. _____ Ba(NO$_3$)$_2$(aq) + _____ NH$_2$SO$_3$H(aq) + _____ H$_2$O(l) $\rightarrow$

   _____ Ba(NH$_2$)$_2$(s) + _____ H$_2$SO$_4$(aq) + _____ HNO$_3$(aq)

2. Suppose you had 0.70 g of Ba(NO$_3$)$_2$ and 1.24 g of NH$_2$SO$_3$H, which would be the limiting reactant in reactions (a), (b) and (c)? Is the limiting reactant the same for all three? (0.7)

3. Calculate the molar mass of magnesium sulfate heptahydrate, MgSO$_4$$\cdot$7H$_2$O(s). (0.3)
Experiment 10 – Part I: Data, Calculations and Results

Organize your data and calculations in the clearest and neatest way that you see fit. Be sure to include the proper number of significant figures and units!!

Data
Record the mass of each reactant, the mass of filter paper and the final mass of your product. (0.5 pt)

Calculations
**Goal:** The goal of the calculations is to determine which of the three reactions occurred. For each of the three reactions, calculate the theoretical yield of the solid product. Do this by finding the moles of each reactant, determining the limiting reactant, and using stoichiometry to find the amount of your solid product. *Remember to clearly present your calculations, and clearly indicate your final answer.* (2 pts)

Compare your measured mass of product with the theoretical yield calculated for reactions (a), (b) and (c). What is the identity of your solid precipitate? ____________________________ (0.5 pt)

Briefly explain your reasoning: ____________________________________________ (1 pt)
Experiment 10 – Part II: Data, Calculations and Results

Organize your data and calculations in the clearest and neatest way that you see fit. Be sure to include the proper number of significant figures and units!!

**Data**
Record the mass of the empty test tube, the mass of the test tube + reactant, and the final mass of the test tube + product here: (0.5 pt)

_____________________________________________________
_____________________________________________________
_____________________________________________________
______________________________________________________________

**Calculations**
**Goal:** The goal of the calculations is to determine how many water molecules are present per CuSO₄ formula unit. You should be able to do so stepwise through the determination of: (i) the mass of water driven off, (ii) the moles of water driven off, (iii) the moles of anhydrous copper(II) sulfate, CuSO₄(s), present in the product. (2 pt)

The experimentally determined molecular formula of the hydrated copper(II) sulfate solid is __________

Briefly explain your reasoning: _____________________________________________________________

________________________________________________________________________________________
________________________________________________________________________________________
________________________________________________________________________________________
________________________________________________________________________________________
________________________________________________________________________________________
(1.5 pt)
Experiment 11A – Separation of Ink by Paper Chromatography

Goal: To observe the separation of components in a mixture in paper chromatography.

Objectives
1. Detail the theory of paper chromatography.
2. Calculate the $R_f$ values for components of a separated mixture.
3. Define: chromatography, mobile phase, stationary phase
4. Given two substances and their relative chemical affinities for the mobile phase and stationary phase, indicate (a) which substance travels further along the chromatography paper, (b) which substance has the greater $R_f$ value
5. Given which of two substances travels further along the chromatography paper, indicate the substance has the greater relative chemical affinity for the mobile phase and which has the greater relative chemical affinity for the stationary phase.
6. Draw a diagram of the main components of a gas chromatograph. Explain its use and how it operates.

Introduction
The separation, detection, and identification of the components of a mixture can be accomplished by several techniques. Each of these techniques depends on the differing chemical or physical properties of the components of the mixture. Chromatography is one such technique. Paper chromatography, which is used here, is just one of several chromatographic methods available.

In paper chromatography, a concentrated drop of solution containing a mixture of substances is placed near one end of a rectangular piece of chromatography paper. The paper will serve as the stationary phase. The end of the paper is immersed in a liquid to a point that is just below the spot where the drop was placed on the paper. The liquid is the mobile phase. Capillary action (the same phenomenon that causes water to travel up a bath towel when an edge of the towel is immersed) causes the liquid to flow up the filter paper. When the liquid reaches the spot, the components of the mixture will begin to migrate upward with the mobile phase. Each component will have a characteristic chemical affinity for the stationary phase and a characteristic chemical affinity for the liquid. These affinities are competitive: The component's affinity for the stationary phase tends to hold the component in one place, but its affinity for the mobile phase tends to make the component follow the liquid as it moves upward. A component with a strong affinity for the stationary phase and a weak affinity for the mobile phase will move more slowly than a component with a weaker affinity for the stationary phase and a stronger affinity for the mobile phase.

A substance’s relative affinity for the stationary and mobile phases is entirely characteristic of that substance. Different substances will have different competitive affinities. In large part, the affinity between a substance and the mobile and/or stationary phases is determined from polarity considerations. Because each component of a mixture will have its own characteristic affinity, each component will travel up the paper at its own characteristic rate. If the stationary phase is sufficiently large, all the components can be separated by the time the mobile phase has reached the top of the stationary phase.
Each component will now appear as a separate spot, unless two components happen to have equal relative affinities for the mobile and stationary phases. If the components are highly colored, the spots will be visible. One can convert weakly colored or colorless spots to highly colored spots by spraying them with substances that react with the components in the spots. The stationary phase will now contain a vertical array of colored spots arranged according to their characteristic rates of ascent. The word chromatography, which is derived from two Greek words and literally means “written in color,” was coined to describe this phenomenon.

The distance traveled by a component of a spot with respect to the distance traveled by the pure liquid is a measure of that component's competitive affinities for the stationary and mobile phases. We define the component's R<sub>f</sub> (retention factor) value in those terms:

\[
R_f = \frac{\text{distance traveled by spot}}{\text{distance traveled by mobile phase}}
\]

The R<sub>f</sub> value of a substance is characteristic of that substance and should be a constant under invariant experimental conditions. The largest R<sub>f</sub> value that any component can possibly have is 1.

Concept of the experiment
The paper chromatography of commercial inks will be examined. The laboratory instructor will assign each partner group four separate ink samples for analysis, and a mobile phase (distilled water, acetone, toluene, or denatured ethanol). One group will use acetone, one toluene, one denatured ethanol, and all other groups will do distilled water.

CAUTION: Because of the volatility and flammability of acetone and toluene, no flames will be allowed in the lab.

The R<sub>f</sub> value for each component of the ink sample will be determined by observing its ascent in the absence of the other substances.

Procedure
1. Obtain a 4×4 inch piece of chromatography paper and a 600 mL beaker.
2. Pour (or pipet) some of the mobile phase into 600 mL beaker. The height of the mobile phase in the beaker should be slightly less than ~0.5 inch.
3. Using a pencil, lightly draw a line across the paper 0.75 inches away from the edge. This is the bottom edge of the paper, and the line marks the starting points for the four chromatograms. It is important that the pencil line be above the level of liquid in the beaker. The liquid must be pulled up the paper and then travel through the starting points.
4. Fold the paper in half so that the line is bisected. In the same manner, fold the paper in half a second time. The paper is now divided into four 1×4 inch sections.
5. At the top of each panel, label each ink sample (brand, permanent or non-permanent, color) using pencil.
6. Take one of the ink pens and make a small mark on the pencil line in the center of one of the panels.
7. Allow the spot to dry. The maximum diameter of an acceptable spot is no larger than 0.25 cm.
8. Gently place the folded paper inside the beaker. The pencil line and the spots must be above the surface of the liquid. Do not splash or allow the paper to be in contact with the sides of the beaker.

9. The beaker must be absolutely stationary throughout the experiment. Do not move the beaker until the run has concluded.

10. Allow the liquid to ascend to within 1.0 inch from the top of the paper, but not to the top. For acetone, only about 5 minutes is required. Toluene takes about 8 minutes, while water and ethanol take longer.

11. When the liquid has reached the desired height, remove the paper from the beaker. Place the wet paper on a paper towel and mark, with a pencil, the position to which the mobile phase has ascended. Work quickly if the mobile phase is acetone or toluene, as these solvents are volatile.

12. Measure and record the vertical distance from the pencil line that the mobile phase has ascended for all spots in each chromatogram. Because the ink samples will streak (smear), be consistent by always marking to the top of the spot (the greatest each color traveled) for each ink component. Calculate the R_f value for every spot.
Experiment 11 – Pre-laboratory Assignment

Name: ___________________________  Lab Day and Time: ___________

1. Suppose there are two substances, A and B. For which of the following two possibilities would you expect there to be the largest forces of attraction between A and B?

   (a) Substance A is polar and substance B is polar.

   (b) Substance A is polar and substance B is nonpolar.

   Explain your choice:


2. Define the following terms. Give generalized definitions, not definitions that are specific for today’s experiment. For example, a definition of stationary phase is NOT: “the paper.”

   (a) mobile phase

   (b) $R_f$ factor

   (c) unit cell
Experiment 11A – Report Sheet

Name: _________________________________        Lab Partner: __________

1. The mobile phase used in this group was ______________.
2. For the four ink samples that were analyzed, complete the following chart.

<table>
<thead>
<tr>
<th>Ink Sample</th>
<th>Observations</th>
<th>Distances</th>
<th>$R_f$ Value</th>
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</table>

CHEM 132 – Experiment 11
Experiment 11A – Post-laboratory Questions

Name: ________________________________  Lab Partner: ______________

1. Why is a pencil used to draw the lines on the piece of chromatography paper rather than an ink pen?

2. Which of the solvents used are primarily polar? Which type of ink (permanent or water soluble) do you believe contains polar components?

3. A piece of chromatography paper is spotted with a solution containing a mixture of two components, A and B. The chemical affinity of A for the stationary phase is greater than that of B, and the chemical affinity of A for the mobile phase is less than that of B. Which substance will have traveled further at the completion of the chromatography experiment? Which substance will have the larger $R_f$ value? Explain.

4. In groups of four or five, have the laboratory instructor show the department’s gas chromatograph. Draw a diagram of the major components of the instrument. Explain in your own words how the instrument works and its function.
Experiment 1B – Structures of Solid State Materials

Goal: To use solid state models to gain a fundamental understanding of crystalline structure.

Objectives
1. Understand the concept of a unit cell.
2. To identify and construct models of (a) Primitive cubic unit cells, (b) Body-centered cubic unit cells, (c) Face-centered cubic unit cells.
3. To determine the stoichiometry of a cubic crystalline solid given the structure of its unit cell.

Introduction
A solid is classified as crystalline if there exists a periodic repetition of its structure in three-dimensional space. The structure that repeats itself throughout space may be an atom or molecule, a group of atoms or ions, or a group of molecules. The smallest possible three-dimensional representation of the periodic structure is called the unit cell. All crystalline solids can be classified using unit cells that fit into 7 basic crystal systems. The most symmetric of these is the cubic system, in which the unit cell possesses a cubic shape.

Within the cubic system, there exist three different sub-classes of unit cells. These sub-classes differ in the arrangement within the cube of their lattice points. Crystal lattice points describe identical structural positions in a solid. The simplest crystalline solids (and all of those we will be investigating today) have individual atoms or ions located at their lattice points. The cubic system subclasses and their descriptions follow:
(a) Primitive (or simple) cubic – Lattice points are located at all corners of the cube.
(b) Body-centered cubic – Lattice points are located at all corners of cube and in the center of the cube.
(c) Face-centered cubic – Lattice points are located at all corners of the cube and in the center of each of the six faces of the cube.

The last of these, face-centered cubic (fcc), is also known as cubic closest packing. This is because it describes the most efficient way possible to fill three-dimensional space with spheres of a given size. In many crystalline ionic compounds, cations or anions or located at the lattice points. In most cases, it is useful to describe the unit cell with the anions at the lattice points because they are typically larger than the cations. In such a description, there exist spaces in the structure into which the smaller ions, typically the cations, can fill. In the fcc unit cell, there are two types of spaces available to be filled. These are the octahedral holes and the tetrahedral holes. The octahedral holes are located along each of the twelve edges of the cube and one is in the center of the cube itself. The tetrahedral holes are located in the middle of the tetrahedron formed in between each corner lattice point and its adjacent three face-centered lattice points.

Because the unit cell is representative of the entire crystalline structure, the stoichiometry of the unit cell will be equivalent to the stoichiometry of the bulk solid. However, determining the stoichiometry of the unit cell is not as simple as counting the numbers of each type of atom or ion that is used to build the cell. The reason for this is that an atom or ion that is located at the corner, on the face, or along the edge of a cell is shared between several unit cells. In fact, an atom or ion at the corner of a cubic unit cell is shared by a total of eight unit cells! For this reason, only a fraction of the atoms or ions at the corners, on the faces, or along the edges may count toward the stoichiometry of the given unit cell. The fraction that each type of lattice point counts toward the unit cell stoichiometry is given below:
<table>
<thead>
<tr>
<th>Lattice Point</th>
<th>Fraction Counted Toward Unit Cell Stoichiometry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corner</td>
<td>$\frac{1}{8}$</td>
</tr>
<tr>
<td>Edge</td>
<td>$\frac{1}{4}$</td>
</tr>
<tr>
<td>Face</td>
<td>$\frac{1}{2}$</td>
</tr>
<tr>
<td>Body</td>
<td>1</td>
</tr>
</tbody>
</table>

In this lab, a variety of crystalline solid unit cells will be built using the solid-state model kits, and a series of questions about the unit cells and the stoichiometry of the solids represented will be answered.
Name: ________________________________

**Experiment 11B – Questions**

1. Using the template with an “A” in the upper left hand corner, build a simple cubic unit cell using the clear spheres. How many clear atoms are present in the unit cell? Remember to take into account the fraction of each atom that is contained in your unit cell.

2. Rebuild the primitive unit cell but with a green sphere in the center of the cube. If the green sphere represents Cs\(^+\) and the clear spheres represent Cl\(^-\), what is the stoichiometry of the cesium chloride unit cell?

3. Construct a body-centered cubic cell using the template with a “B” in the upper left-hand corner and the clear spheres. How many clear atoms are present in the unit cell?

4. Remove all of the metal rods from the “A” template and replace it with the “C” template. Place nine metal rods in the shaded area. Construct a face-centered cubic cell in this template using the clear spheres. (Hint: it requires 14 clear spheres). How many clear atoms are present in the unit cell?

5. Rebuild the fcc unit cell with blue spheres in all of the octahedral holes. If the blue spheres represent Na\(^+\) and the clear spheres represent Cl\(^-\), what is the stoichiometry of the sodium chloride unit cell?

6. Rebuild the fcc unit cell once again but remove all of the blue spheres. Lift up on one of the corner clear spheres and place a pink sphere into the tetrahedral hole. (Be careful - it will not be on a metal rod, yet it should fit snugly in place). Suppose that four of the possible eight tetrahedral holes in the model were filled. If the pink spheres represent Zn\(^{2+}\) and the clear spheres represent S\(^2-\), what is the stoichiometry of the zinc sulfide unit cell?
Experiment 12 – The Determination of the Molarity of an Unknown Cobalt(II) Chloride Solution by Visible Photometry

*Goal:* To determine the molarity of a cobalt(II) chloride solution of unknown concentration.

**Objectives**
1. Define: Beer’s Law, frequency, wavelength, molarity
2. Given a solid solute and water, prepare a specific volume of an aqueous solution to a specified molarity
3. Given a solution of a particular molarity, dilute the solution (or a portion of the solution) to a specified volume and determine its new molar concentration
4. Use Beer’s Law to determine the expected absorbance of a solution of known concentration given the equation of the appropriate calibration curve.

**Introduction**

When gaseous substances are heated (thermal excitation), they eventually emit light of characteristic frequencies (emission spectra). If exposed to electromagnetic radiation, gaseous substances will also absorb characteristic frequencies of light (absorption spectra). This process is exactly the opposite of emission.

During the absorption of light, an electron undergoes a transition from a lower energy level to a higher energy level. The electron gains energy in this process by absorbing a photon, whose energy \( E \) is given by:

\[
E = h \nu
\]

The energy of the photon corresponds to the difference in energy between the higher and lower energy levels. As a result of the transition, light having the frequency \( \nu \) is absorbed while other frequencies are transmitted through the sample.

A very important concept in analytical spectroscopy is known as **Beer’s Law**, which states that the amount of light absorbed \( A \) by a solute in solution will be directly proportional to the concentration of that solute in solution:

\[
A \propto M
\]

where \( M \) is the molarity of the solution.

If the concentration of the solute in solution is cut in half by dilution, the absorbance of the solution should be cut in half as well. Before using this absorption property, the calculation of the concentration of a solution after dilution must be understood:

\[
M_{\text{conc}} V_{\text{conc}} = M_{\text{dil}} V_{\text{dil}}
\]
Concept of the Experiment

Known: The absorbance of a 0.150 M solution of CoCl₂ will be measured before and after a series of dilutions. For each dilution, calculate the new concentration of the CoCl₂ solution by using

\[ M_{\text{conc}} \times V_{\text{conc}} = M_{\text{dil}} \times V_{\text{dil}} \]

Because absorbance is directly proportional to concentration, the equation for the best-fit straight line for a graph in which absorbance (A) is plotted (y-axis, ordinate) against the concentration (M) of the CoCl₂ aqueous solutions (x-axis, abscissa) will take the form

\[ y = mx + b \]

The equation of the best-fit line will be determined by linear regression by using spreadsheet software to give a calibration curve under the experimental conditions.

Unknown: The calibration curve calculated above will be used to determine the concentration of an unknown CoCl₂ solution. The unknown concentration is too large for an accurate measurement from the absorption of light and will therefore need to be diluted.

Before measuring any of these absorbencies, however, one will need to find the correct wavelength for the measurements. This wavelength is the wavelength in the absorption spectrum of CoCl₂ at which the maximum absorbance occurs. Use of this wavelength will allow maximum sensitivity for each measurement.

Important Notes

- All solutions will be discarded in an inorganic waste container in the laboratory. Nothing goes down the sink drain, as cobalt is harmful.
- Obtain guidance for using the Spec-20 spectrophotometer.

Procedure

1. Rinse and dry 8 spectroscopy-grade test tubes, and then place them in a test tube rack.
2. Use burets to make the additions of 0.150 M CoCl₂ and distilled water shown in Table 12.1. Thoroughly mix the contents of each test tube. Do not use one of your fingers as a stopper.

Table 12.1 Dilutions for known cobalt(II) chloride solutions.

<table>
<thead>
<tr>
<th>Test Tube No.</th>
<th>0.150 M CoCl₂ Solution (mL)</th>
<th>Distilled Water (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0</td>
<td>5.0</td>
</tr>
<tr>
<td>2</td>
<td>1.0</td>
<td>4.0</td>
</tr>
<tr>
<td>3</td>
<td>2.0</td>
<td>3.0</td>
</tr>
<tr>
<td>4</td>
<td>2.5</td>
<td>2.5</td>
</tr>
<tr>
<td>5</td>
<td>3.0</td>
<td>2.0</td>
</tr>
<tr>
<td>6</td>
<td>3.5</td>
<td>1.5</td>
</tr>
<tr>
<td>7</td>
<td>4.0</td>
<td>1.0</td>
</tr>
<tr>
<td>8</td>
<td>5.0</td>
<td>0.0</td>
</tr>
</tbody>
</table>
3. Set up the spectrometer to read percent transmittance (%T). Calibrate the spectrometer in two steps. First, with the sample lid closed and no sample in the holder, turn the 0 %T control knob until it reads 0.0. Second, with a clean test tube filled with distilled water in the holder, turn the 100 %T knob until it reads 100.0.

4. Using the sample in test tube 8, obtain the %T of aqueous CoCl₂, measuring the percent transmittance at intervals of 25 nm between 425 nm and 600 nm. Do not dispose of this solution. Record the results in the Data Table.

5. From these measurements, select the wavelength at which the %T is smallest (and hence the absorbance is the largest). This wavelength will provide maximum sensitivity for the detection of Co²⁺ ion.

6. Set the spectrometer at the maximum sensitivity wavelength determined in Step 5 and recalibrate according to Step 3. Use this wavelength for all subsequent measurements.

7. Measure the %T of test tube 1 at the set wavelength. Measure the %T of the contents of the remaining test tubes (2–8). Record all results in a data table.

8. Using a 10 mL graduated cylinder, obtain a 5.0 mL sample of the unknown solution and measure the %T using the same wavelength in Step 6. Record this %T value. Then, record the %T of a solution prepared by mixing 4.0 mL of distilled water with 1.0 mL of the unknown. See if either of these %T values falls in the range of the values recorded for the eight known solutions. If neither is in that range, try another proportion of unknown plus water. Construct a data table in the “Unknown Determination” section that includes all of the diluted solutions. Keep diluting until you obtain a mixture such that the %T of the solution lies within the range of %T values that were measured in Step 6.

9. The %T and absorbance (A) are related through: \( A = -\log(T) \). Convert all of the %T values from the data table to absorbance (A) values. As an example, if the sample has a %T = 50, then \( T = 0.50 \) and \( A = -\log(0.50) = 0.30 \).

10. Enter the absorbance vs. concentration measurements into Excel in the computer lab. Obtain the equation of the linear regression line that fits the data. Do not include the unknown solution on the graph.

11. Calculate the molar concentration of CoCl₂ in the diluted unknown solution using the absorbance of the diluted unknown solution and the equation for the best-fit line.

12. Calculate the concentration of CoCl₂ in the original, undiluted unknown using the dilution formula (if the unknown solution was not diluted in Step 8, this will be identical to Step 11).
Experiment 12 – Pre-laboratory Assignment

Name: ________________________________  Lab Day and Time: ______________

1. What is Beer’s Law?

2. The following data were collected when the absorbencies of a series of solutions containing NiCl$_2$ were measured in the laboratory.

<table>
<thead>
<tr>
<th>Test Tube Number</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volume (mL) of 0.390 $M$ NiCl$<em>2$ stock solution ($V</em>{conc}$)</td>
<td>4.0</td>
<td>3.0</td>
<td>2.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Volume (mL) of H$_2$O added</td>
<td>0.0</td>
<td>1.0</td>
<td>2.0</td>
<td>3.0</td>
</tr>
<tr>
<td>Concentration ($M$) of resulting diluted NiCl$<em>2$ solution ($M</em>{dil}$)</td>
<td>0.390</td>
<td>[ ]</td>
<td>[ ]</td>
<td>[ ]</td>
</tr>
<tr>
<td>Absorbance (A)</td>
<td>0.858</td>
<td>0.644</td>
<td>0.429</td>
<td>0.215</td>
</tr>
</tbody>
</table>

Complete the table by calculating the concentration of NiCl$_2$ in each diluted solution. To do this, use the dilution equation; show your work on the back if necessary:

$$M_{conc}V_{conc} = M_{dil}V_{dil}$$

The volume of the dilute solution is obtained in each case by adding the volume of the 0.390 $M$ solution ($V_{conc}$) and the volume of water. You can see that the volume of the dilute solution ($V_{dil}$) is always 4.0 mL. Calculate the concentration of the dilute solution in each case. The molarity of the concentrated solution ($M_{conc}$) is 0.390 $M$. Show all calculations below.
Experiment 12 – Data

1. The Percent Transmission Spectrum of Aqueous CoCl₂

<table>
<thead>
<tr>
<th>λ, nm</th>
<th>%T</th>
<th>λ, nm</th>
<th>%T</th>
</tr>
</thead>
<tbody>
<tr>
<td>425</td>
<td></td>
<td>525</td>
<td></td>
</tr>
<tr>
<td>450</td>
<td></td>
<td>550</td>
<td></td>
</tr>
<tr>
<td>475</td>
<td></td>
<td>575</td>
<td></td>
</tr>
<tr>
<td>500</td>
<td></td>
<td>600</td>
<td></td>
</tr>
</tbody>
</table>

The minimum percent transmission occurs at ________ nm.

2. Create a data table including all 8 of your CoCl₂ solutions. For each sample, include the %T, the value of T (where T = %T/100), the absorbance (A = –log T) and the concentration in units of moles per liter (M). As a sample, show the calculations for all of these values for test tube #2 on the following page.
Experiment 12 – Calculations

Show the calculations for the values corresponding to test tube 2 entered in the data table on the previous page. Include calculations for all of the test tubes if preferred.
Experiment 12 – Unknown Determination

Data
Give the equation of the best-fit line generated by linear regression, in the form $A = \text{slope} \times M$ and your unknown number.

Unknown No. _______

Construct a data table below that includes all mixtures that were made of the unknown from step 8. There should be at least two data points (the 5.0 mL unknown + 0.0 mL water and the 1.0 mL unknown + 4.0 mL water). For each solution, include the %T and the absorbencies.

Calculations and Results
Show all calculations for the determination of the molarity of the unknown solution. Present them in a clear, well-organized manner.

Unknown Concentration: _____________________
Experiment 12 – Post Lab Questions

1. How many grams of solid CoCl\(_2\) should be dissolved to make 300.0 mL of a 0.183 \(M\) solution?

2. Using the 0.183 \(M\) CoCl\(_2\) solution above, how much water must be added to make a 0.069 \(M\) solution?

3. Suppose that a 0.150 \(M\) solution is known to give a %T value of 56.3%. Under the same conditions, another solution results in a %T of 32.3%. Using Beer’s Law, determine the approximate concentration of the second solution.
Experiment 13 – The Determination of a Rate Law

**Goal:** Determine the rate law for a reaction by measuring concentrations versus time and using the method of initial rates.

**Objectives**
1. Be able to give the form of a rate law
2. Use the method of initial rates to determine a rate law
3. Calculate a rate constant
4. Determine the rate of a reaction using graphical analysis

**Introduction**

The rates at which reactions occur vary greatly. The study of reaction rates and the factors that influence them is called **kinetics**. The rate of a reaction is defined as the change in concentration of a species involved in a reaction with respect to time. For example, in the general reaction:

\[ \text{A} + 2 \text{B} \rightarrow 3 \text{C} + \text{D} \]

the rate could be defined as:

\[ \text{rate} = \frac{\Delta [\text{A}]}{\Delta \text{time}} \]

The rate could also be defined with respect to \(\Delta [\text{B}], \Delta [\text{C}] \text{ or } \Delta [\text{D}]\). In order to avoid ambiguity, the rate is most often defined as the concentration change of a species with respect to time, divided by the coefficient of that species in the balanced equation. The rate is made positive by using a negative sign with reactants.

\[ \text{rate} = -\frac{\Delta [\text{A}]}{\Delta \text{time}} = -\frac{\Delta [\text{B}]}{2(\Delta \text{time})} = \frac{\Delta [\text{C}]}{3(\Delta \text{time})} = \frac{\Delta [\text{D}]}{\Delta \text{time}} \]

Thus, the rate for our reaction above is independent of which species is chosen to define it. Among the factors that influence the rates of a reaction are the concentrations of the reactants. The dependence of the rate of reaction on the concentrations of the reactants can be expressed mathematically in terms of a rate law. The most general form of the rate law for the reaction above is:

\[ \text{rate} = k[\text{A}]^X[\text{B}]^Y \]

where \(k\) is the rate constant, and superscripts \(X\) and \(Y\) are the orders of the reaction with respect to \(A\) and \(B\) respectively.

One way to determine the values of \(k, X\) and \(Y\) is through the method of initial rates. In this method, the rate of reaction is measured for several different initial concentrations of reactants. By comparing the rates of reaction, the values of \(X\) and \(Y\) can be calculated. Once they are known, the value of \(k\) can also be determined. The method of initial rates will be used in this experiment to determine the rate law for a reaction.
Concept of the experiment

The following reaction will be investigated:

\[ \text{S}_2\text{O}_8^{2-} + 2\Gamma \rightarrow \text{I}_2 + 2\text{SO}_4^{2-} \]  

(1)

As suggested by the discussion in the introduction, the form of the rate law can be written with respect to the concentrations of the reactants. Thus, the rate law can be written in the form:

\[ \text{rate} = k[\text{S}_2\text{O}_8^{2-}]^X[\Gamma]^Y \]  

(2)

The goal of this laboratory is to determine the values of \( k \), \( X \), and \( Y \), and therefore be able to write the actual rate law for Reaction (1).

A color change will accompany the progress of Reaction (1). When \( \text{I}_2 \) (a product of the reaction under study) and starch (which will be added to the reaction container) are present, a starch-iodine complex is observed to give a blue-black color. Therefore, the progress of the reaction, and formation of \( \text{I}_2 \), can be observed by noting the appearance of this color.

Yet to determine a rate, more information than the appearance of a product must be known. As discussed in the introduction, the change in the amount of a product or reactant must also be measured. To enable this measurement, a known amount of thiosulfate ion, \( \text{S}_2\text{O}_3^{2-} \), will be added to the reaction container. Do not confuse the \( \text{S}_2\text{O}_3^{2-} \) and \( \text{S}_2\text{O}_8^{2-} \) ions in this discussion. The thiosulfate ion reacts with \( \text{I}_2 \) as follows:

\[ \text{I}_2 + 2\text{S}_2\text{O}_3^{2-} \rightarrow 2\Gamma + \text{S}_4\text{O}_6^{2-} \]  

(3)

The presence of the thiosulfate ion will therefore consume any \( \text{I}_2 \) produced, masking the appearance of the blue-black color mentioned above.

When a known amount of \( \text{S}_2\text{O}_3^{2-} \) has been reacted, any more \( \text{I}_2 \) produced will result in a color change. Using stoichiometry with Reactions (1) and (3), it is easy to see that if a known amount of \( \text{S}_2\text{O}_3^{2-} \) is reacted:

\[ \text{mol I}_2 \text{ reacted in Rxn (3)} = \frac{1}{2} \times \text{mol S}_2\text{O}_3^{2-} \text{ reacted in Rxn (3)} \]

\[ \text{mol I}_2 \text{ produced in Rxn (1)} = \text{mol I}_2 \text{ reacted in Rxn (3)} \]

\[ \text{mol S}_2\text{O}_8^{2-} \text{ reacted in Rxn (1)} = \text{mol I}_2 \text{ produced in Rxn (1)} \]

The net result is:

\[ \text{mol S}_2\text{O}_8^{2-} \text{ reacted} = \frac{1}{2} \times \text{mol S}_2\text{O}_3^{2-} \text{ reacted in Rxn (3)} \]

In this experiment, the amount of \( \text{S}_2\text{O}_8^{2-} \) reacted as a function of time will be determined by observing how much \( \text{S}_2\text{O}_3^{2-} \) has reacted. The above equalities will be used to indirectly relate \([\text{S}_2\text{O}_3^{2-}]\) to \([\text{S}_2\text{O}_8^{2-}]\).

This laboratory will require two weeks to complete. In the first week, Reaction (1) will be observed as a function of initial reactant concentrations. Use the method described above in order to determine the amount of \( \text{S}_2\text{O}_8^{2-} \) reacted as a function of time. During the second week of the lab, analyze the rates of the reaction as a function of the initial reactant concentration. Then use the method of initial rates to determine the rate law for the reaction.
Plotting the amount of $S_2O_8^{2-}$ reacted versus the elapsed reaction time allows the rate of the reaction calculation. The slope of this line is the rate of change in the moles of $S_2O_8^{2-}$. In order to determine the rate in terms of the molarity, it must be divided by the total volume of the solution in L.

$$slope = \frac{\Delta[S_2O_8^{2-}]}{\Delta t}$$

(determined from linear regression)

$$rate = \frac{\text{slope}}{\text{volume(L)}}$$

Two other compounds are necessary, but do not contribute to the rate law:
- KNO$_3$ is added as a counter-ion to maintain the same ion concentration throughout all of the trials. It does not participate in the reaction.
- The reaction is catalyzed by metal ions. A drop of a 0.10 M EDTA solution is added to form metal complexes and reduce any catalytic effects of trace metal ions in the reaction samples.

The rates of reaction by this procedure will be determined using four different reactant concentrations. These four different conditions will be investigated separately in Trials 1, 2, 3, and 4. The rate will be determined for each. The method of initial rates will be employed to determine the overall rate law.

**Procedure**

**CAUTION: Discard all solutions in an inorganic waste container.**

1. The solutions that will be prepared for each of the four trials are given in Table 1. The source of the first reactant, $S_2O_8^{2-}$, is 0.20 M $(NH_4)_2S_2O_8(aq)$. Using a buret, prepare this solution for Trial 1 in a 100 mL beaker.

2. The source for the second reactant, I$^-$ is 0.20 M KI($aq$). This solution should be mixed with the starch, the first aliquot of $S_2O_3^{2-}$, the KNO$_3$ counter-ions, and EDTA in a 250 mL Erlenmeyer flask.

3. Using a buret for the KI, prepare this solution now for Trial 1. The first aliquot of $S_2O_3^{2-}$ is provided by 0.40 M $S_2O_3^{2-}$.

4. Fill each of seven test tubes with 1.0 mL of Na$_2$S$_2$O$_3$. Keep them in a test tube rack nearby. Be prepared by reading ahead through steps 5–7 now.

5. The reaction is now ready to begin. Be ready to start the timer and to work quickly. The reaction may be rather fast. Start the timer and add the contents of the 100 mL beaker, the $(NH_4)_2S_2O_8$, to the Erlenmeyer flask. Swirl the flask after mixing and every few seconds thereafter.

6. Watch carefully for the color change. Two things must be done as soon as the color changes. The first is to record the time elapsed on the timer, but do not stop it! The second is to add the contents of the next one of the seven test tubes and swirl the flask. It should be decided beforehand which person is responsible for time-keeping and which person is responsible for adding the test tubes. Work Quickly!
7. Repeat step 6 until all seven of the test tubes have been added and all 8 times have been recorded for the trial on the data sheet. Each data point should be recorded in seconds from the initial time of mixing in step 4.

**Note:** With each color change, $4.0 \times 10^{-4}$ moles of $S_2O_3^{2-}$ have reacted. Thus, this means that $2.0 \times 10^{-4}$ moles of $S_2O_8^{2-}$ have reacted. For each trial, the times for eight color changes will be recorded, each indicating that an additional $2.0 \times 10^{-4}$ moles of $S_2O_8^{2-}$ have reacted. Data for the amount of $S_2O_8^{2-}$ reacted versus time should now be present.

8. When completed, reset the timer and empty the flask in the waste container.

9. Prepare the next trial using the amounts listed in Table 1 below. Repeat Steps 1 – 7 until all four trials have been completed.

### Table 1. Amounts of each component needed for each trial.

<table>
<thead>
<tr>
<th></th>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
<th>Trial 4</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Contents of 100 mL beaker</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$(NH_4)_2S_2O_8$</td>
<td>25.0 mL</td>
<td>50.0 mL</td>
<td>25.0 mL</td>
<td>50.0 mL</td>
</tr>
<tr>
<td><strong>Contents of 250 mL Erlenmeyer flask</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$25.0$ mL KI</td>
<td>25.0 mL KI</td>
<td>50.0 mL KI</td>
<td>12.5 mL KI</td>
<td></td>
</tr>
<tr>
<td>$1.0$ mL starch</td>
<td>1.0 mL starch</td>
<td>1.0 mL starch</td>
<td>1.0 mL starch</td>
<td></td>
</tr>
<tr>
<td>$1.0$ mL $Na_2S_2O_3$</td>
<td>1.0 mL $Na_2S_2O_3$</td>
<td>1.0 mL $Na_2S_2O_3$</td>
<td>1.0 mL $Na_2S_2O_3$</td>
<td></td>
</tr>
<tr>
<td>$48.0$ mL KNO$_3$</td>
<td>23.0 mL KNO$_3$</td>
<td>23.0 mL KNO$_3$</td>
<td>35.5 mL KNO$_3$</td>
<td></td>
</tr>
<tr>
<td>1 drop EDTA</td>
<td>1 drop EDTA</td>
<td>1 drop EDTA</td>
<td>1 drop EDTA</td>
<td></td>
</tr>
<tr>
<td><strong>Total volume of reaction solution</strong></td>
<td>100 mL</td>
<td>100 mL</td>
<td>100 mL</td>
<td>100 mL</td>
</tr>
<tr>
<td><strong>Additional Requirements</strong></td>
<td>7 × 1.0 mL aliquots of $Na_2S_2O_3$</td>
<td>7 × 1.0 mL aliquots of $Na_2S_2O_3$</td>
<td>7 × 1.0 mL aliquots of $Na_2S_2O_3$</td>
<td>7 × 1.0 mL aliquots of $Na_2S_2O_3$</td>
</tr>
</tbody>
</table>
Calculations

1. Complete the data sheet. For each trial, make a data table that contains the 8 data points. These data points should have the amount of time elapsed and the moles of $S_2O_8^{2-}$ consumed in reaction 1. Remember, the moles of $S_2O_8^{2-}$ consumed can be found through the relationship, discussed in the introduction, to the number of moles of $S_2O_3^{2-}$.

2. Make four plots (one for each trial) using Excel. Plot the moles of $S_2O_8^{2-}$ consumed versus the time elapsed from the start of the reaction. Include the (0, 0) point as the first point in the plot. Be sure to include the plots when the lab is submitted for a grade.

3. Determine the rates of the four reactions by performing a linear regression with each of the four plots. The rate is equal to the slope of the line divided by the total volume (0.100 L for each trial). Report the rates of the 4 reactions (with the correct units!) on the report sheet.

4. Determine the initial concentrations of $I^-$ and $S_2O_8^{2-}$ for each trial and report them on the report sheet. To do this, use the dilution formula:

$$M_1V_1 = M_2V_2$$

Remember that each solution has been diluted to 100 mL. The lab will be completed at the beginning of week 2 for this laboratory.

5. Using the method of initial rates determine the factors $X$ and $Y$ in the rate law. Round to the nearest integer if necessary.

6. Calculate the value of $k$ in the rate law. Be sure to include the correct units.
Experiment 13 – Pre – laboratory Assignment

Name: ________________________________ Lab Day and Time: ______________

1. What will be plotted to determine the rates of the observed reactions?

   How many different plots will be made?

2. Determine, using stoichiometry and Reactions 1 and 3 in the lab discussion, how many moles of $S_2O_8^{2-}$ have been consumed after the solution containing 1.0 mL of 0.40 $M$ $S_2O_3^{2-}$ has reacted. (Show all work.)

3. The form of the rate law to be determined is

   \[ \text{rate} = k[S_2O_8^{2-}]^x[I]_y \]

   What three variables must be determined in order to write the rate law?
Experiment 13 – Data

For each of the four trials, create a data table for the results. For each trial, include (1) the total volume of \(S_2O_3^{2-}\) added, (2) the number of moles of \(S_2O_8^{2-}\) consumed, and (3) the cumulative time, in seconds, elapsed since the start of the reaction.

See the lab discussion and the pre-lab to determine the relationship between the amount of \(S_2O_3^{2-}\) added and the number of moles of \(S_2O_8^{2-}\) consumed. Note that the first data point for each trial corresponds to 1.0 mL of 0.40 \(M\) \(S_2O_3^{2-}\) and each subsequent data point corresponds to an additional 1.0 mL.

Trial 1

Trial 2

Trial 3

Trial 4
Experiment 13 – Calculations and Report Sheet

1. Reaction rates determined from plots. See the procedure for the initial concentrations of reactants. Be sure to include correct units.

<table>
<thead>
<tr>
<th>Trial Number</th>
<th>Rate</th>
<th>Initial $[S_{2}O_{8}^{2-}]$</th>
<th>Initial $[I^{-}]$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2. Calculation of reaction order. Calculate $X$ and $Y$ using the method of initial rates. Round to the nearest integer values.

$X = \_\_\_\_$

$Y = \_\_\_\_$

3. Calculation of the rate constant. Calculate $k$ using the correct units.

4. The rate law. Write the complete, experimentally determined rate law.
Experiment 13 – Post Lab Questions

1. State, in each case, whether the reaction rate will increase or decrease.

   a) The concentration of KI is raised: ____________________

   b) The concentration of \((\text{NH}_4)_2\text{S}_2\text{O}_8\) is lowered: ________________

   c) A catalyst is added: ____________________

   d) The temperature is raised: ____________________

2. Using the experimentally determined rate law, calculate the rate of reaction when the concentration of \((\text{NH}_4)_2\text{S}_2\text{O}_8\) is \(3.5 \times 10^{-2} \, M\) and the concentration of KI is \(8.2 \times 10^{-2} \, M\).

3. It is found that for a hypothetical reaction \(A + B \rightarrow C\), that doubling the concentration of A while B is held constant will quadruple the rate of the reaction. Furthermore, doubling the concentration of B while A is held constant doubles the rate of the reaction. Write the rate law for this reaction.
Experiment 14 – The Determination of a Rate Law Using Graphical Analysis

Goal: Use graphical analysis to determine the rate law for a reaction.

Introduction
In general, the rate law for a reaction,

\[ aA + bB \rightarrow eE + fF \]  

(1)

can be expressed as:

\[ \text{rate} = k[A]^x[B]^y \]  

(2)

where \( x \) and \( y \) are the orders of the rate with respect to \( A \) and \( B \), respectively, and \( k \) is the rate constant for the reaction. Note that \( x \) and \( y \) are independent of \( a \) and \( b \). To determine the rate law, we must find the values of \( x \), \( y \) and \( k \).

To proceed, the orders of the rate law with respect to a given reactant can be determined through the analysis of data obtained from a reaction where all other reactant concentrations are held constant.

Consider a case where the concentration of reactant, \( B \), is held constant during a set of experiments. The entire quantity \( k[B]^y \) is now constant and can be rewritten using the notation \( k' \):

\[ k' = k[B]^y \]  

(3)

The notation \( k' \) is now used to distinguish this new quantity from the rate constant \( k \) in equation 2. The benefit of writing the rate law this way is that we will now be able to focus only upon the effect that the concentration of reactant \( A \) has upon the rate law. This is achieved by plotting functions of the concentration of the variable reactant, \([A]\), versus the time. The plot that results in a straight line can be used to determine the order of the reaction with respect to \( A \), and the pseudo rate constant, \( k' \), that satisfies the equation:

\[ \text{rate} = k'[A]^x \]  

(4)

This equation, which only includes the concentration of one reactant, is called a pseudo-rate equation. It has been established that,

1. If \([A] \text{ vs. } t \) is linear: The rate is zero-order (\( x = 0 \)) with respect to \([A]\) and the rate law is: \( \text{rate} = k' \) where the slope = \(-k'\).

2. If \( \ln[A] \text{ vs. } t \) is linear: The rate is first order (\( x = 1 \)) with respect to \([A]\) and the rate law is \( \text{rate} = k'[A] \) where the slope = \(-k'\).

3. If \( 1/[A] \text{ vs. } t \) is linear: The rate is second order (\( x = 2 \)) with respect to \([A]\) and the rate law is \( \text{rate} = k'[A]^2 \) where the slope = \(k'\).

The above treatment suggests how the order of the rate law with respect to reactant \( A \) (we call this \( x \)) and the pseudo rate constant \( k' \) could be found. How is this information then used to find the true rate constant, \( k \), and the overall rate law?
The answer is that a similar analysis must be done by varying [B] and holding [A] constant. In a similar fashion to that discussed above, a pseudo-rate equation can be written as:

\[ \text{rate} = k''[B]^y \]  \hspace{1cm} (5)

where

\[ k'' = k[A]^x \]  \hspace{1cm} (6)

By plotting the data for this reaction, the values for \(y\) and \(k''\) can be found. We now know both \(x\) and \(y\), the reaction orders with respect to \([A]\) and \([B]\).

Using the results for \(x\) and \(y\) and the concentrations of \([A]\) or \([B]\) in the situations where they were held fixed, we can utilize equations (3) and/or (6) to calculate the value of the true rate constant, \(k\). We now know all of the information for the rate law!

**Example**

Suppose that under conditions where \([B]\) was constant and a known value, the reaction was found to be first order with respect to \([A]\). In other words, a plot of ln\([A]\) vs. \(t\) was found to be linear, which means that \(x = 1\) and the slope was equal to \(k'\). We could then write the pseudo-rate equation as:

\[ \text{rate} = k'[A] \]  \hspace{1cm} (7)

Suppose the same analysis was done while holding \([A]\) constant and we found that the reaction was also first order with respect to \([B]\), or \(y = 1\). We could then write:

\[ \text{rate} = k''[B] \]  \hspace{1cm} (8)

Combining equations 2 and 7, with \(y = 1\), gives:

\[ k' = k[B] \]  \hspace{1cm} (9)

Since we know \(k'\) and \([B]\), we can then solve this for \(k\). We now know \(x, y\) and \(k\), and we can write the overall rate law. (\(k\) could also be found by combining equation 2 and 8, with \(x = 1\), since we know \(k''\)).
Experiment 14 – Pre – laboratory Assignment

Name: _______________________________  Lab Date and Time: ______________

1. Suppose that a reaction:

    \[ 2A + B \rightarrow 3C \]

is first order in \([A]\) and \([B]\). Write the general expression for the rate law of the reaction using the rate constant, \(k\).

2. For the above reaction, when \([B]\) is held constant at 0.10 \(M\), the plot of \(\ln[A]\) vs. time gives a straight line, indicating that the reaction is indeed first order in \([A]\). The slope of the line is -2.5 \(s^{-1}\). This means that the pseudo-rate law can be written as:

    \[ \text{rate} = (2.5 \, s^{-1}) \times [A] \]

What is the value of \(k'\) in the above rate expression?

3. Compare the equations in questions 1 and 2 and calculate the value of \(k\).
Procedure

Investigating the reaction:

\[2\text{NO}(g) + \text{O}_2(g) \rightarrow 2\text{NO}_2(g)\]

1. Using the data below, plot $[\text{NO}]$ vs. $t$, $\ln[\text{NO}]$ vs. $t$ and $1/[\text{NO}]$ vs. $t$

2. Determine by inspection if any of the above plots result in a straight line. If a plot results in a straight line, determine the order of the rate with respect to $[\text{NO}]$. In Excel, add a trendline to determine the slope and the $k'$ value for [NO]. (When adding trendline, do not set $b = 0$). The reaction rate can be expressed as:

\[\text{rate} = k'[\text{NO}]^x\]

This $k'$ value is the pseudo-first order rate constant when $[\text{O}_2]$ is constant, but it is not the overall rate constant, k from equation 2.

3. Using the data from experiment 2, where $[\text{O}_2]$ was varied, perform a similar procedure as that described in steps 1 and 2. The plot that results in a straight line can be used to calculate $k''$, where

\[\text{rate} = k''[\text{O}_2]^y\]

4. Determine the value of k with the method described in the introduction.

Experiment 1: Run with $[\text{O}_2] = 8.30 \times 10^{-4} \text{M}$ held constant.

<table>
<thead>
<tr>
<th>Time (s)</th>
<th>$[\text{NO}]$ (M)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>$2.20 \times 10^{-2}$</td>
</tr>
<tr>
<td>5</td>
<td>$1.04 \times 10^{-2}$</td>
</tr>
<tr>
<td>10</td>
<td>$9.91 \times 10^{-3}$</td>
</tr>
<tr>
<td>20</td>
<td>$5.52 \times 10^{-3}$</td>
</tr>
<tr>
<td>30</td>
<td>$4.49 \times 10^{-3}$</td>
</tr>
<tr>
<td>50</td>
<td>$2.62 \times 10^{-3}$</td>
</tr>
<tr>
<td>100</td>
<td>$1.62 \times 10^{-3}$</td>
</tr>
<tr>
<td>150</td>
<td>$9.20 \times 10^{-4}$</td>
</tr>
<tr>
<td>200</td>
<td>$8.56 \times 10^{-4}$</td>
</tr>
</tbody>
</table>

Experiment 2: Run with $[\text{NO}] = 2.20 \times 10^{-2} \text{M}$ held constant.

<table>
<thead>
<tr>
<th>Time (s)</th>
<th>$[\text{O}_2]$ (M)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>$8.30 \times 10^{-4}$</td>
</tr>
<tr>
<td>0.10</td>
<td>$6.19 \times 10^{-4}$</td>
</tr>
<tr>
<td>0.20</td>
<td>$3.82 \times 10^{-4}$</td>
</tr>
<tr>
<td>0.50</td>
<td>$1.01 \times 10^{-4}$</td>
</tr>
<tr>
<td>0.75</td>
<td>$7.42 \times 10^{-5}$</td>
</tr>
<tr>
<td>1.0</td>
<td>$1.85 \times 10^{-5}$</td>
</tr>
<tr>
<td>2.0</td>
<td>$7.91 \times 10^{-7}$</td>
</tr>
<tr>
<td>3.0</td>
<td>$1.54 \times 10^{-8}$</td>
</tr>
<tr>
<td>4.0</td>
<td>$6.63 \times 10^{-10}$</td>
</tr>
</tbody>
</table>
Experiment 14 - Results

Turn in all of the plots and report the rate parameters below.

The rate expressed in terms of [NO]

The rate expressed in terms of [O₂]

\[
\text{rate} = k'[\text{NO}]^x \quad \text{rate} = k''[\text{O}_2]^y
\]

\[
k' = \quad x = \quad k'' = \quad y =
\]

Show calculations for k:

Value for k using \( k' \)  \quad Value for k using \( k'' \)

\[
k = \quad k =
\]

The average value of k

\[
k =
\]

The overall rate law:

\[
\text{rate} =
\]

Post Lab Question

Suppose an equilibrium is established with the reaction in this lab. Write an expression for \( K_C \) for this reaction.
A Strong Acid – Strong Base Titration

Clinton D. Jones, Ph.D.

Purpose

- Learn how to perform a precise and accurate titration by using strong acid-base chemistry
- Learn and understand how to determine an unknown solution concentration using titration calculations
- Gain experience with common equipment for subsequent laboratory titration experiments

Background

The purpose of a titration is to use a solution of known concentration (the titrant) to determine the concentration of an unknown solution (the analyte). (Both the titrant and analyte can be reversible and thus may serve dual roles.) One does this by adding an equal molar amount of titrant to analyte. Theoretically, when the exact moles of titrant equal the exact moles of analyte, the equivalence point is reached. However, what is measured in the laboratory during a titration is called the end point. The end point is slightly different from the true equivalence point, but is well within an acceptable value. Determining the end point instead of the equivalence point is necessary because of a color-changing indicator that cannot change color at the equivalence point, because the indicator itself requires a very slight excess of titrant to change color. As you can tell, some important aspects must be known before planning any titration:

- The chemistry between the titrant and analyte (i.e. how they react together)
- The expected pH range near the equivalence point of the titration
- The accurate concentration of the titrant solution

The Chemistry – Recall from General Chemistry 1 that HCl and NaOH are both strong electrolytes, and thus represent a strong acid and strong base respectively. Both compounds completely dissociate in water, producing Na⁺, H⁺, OH⁻, and Cl⁻ ions. Individual H⁺ and OH⁻ ions will combine with each other to form H₂O in a neutralization reaction. A neutralization reaction is possible with any acid-base combination. However, the simplest case to understand is when both acid and base are strong electrolytes. If both strong acid and strong base are present in the same molar amounts (the equivalence point), then only “salty water” should result, since all acid and base ions have neutralized each other (sodium and chloride ions still remain present). If either the strong acid or strong base is present in a greater amount than the other, then the pH of the solution will deviate from neutrality (7.00).

The Indicator – Today’s experiment requires the colorimetric indicator, phenolphthalein. This compound is colorless in acidic conditions; however, it turns pink under the slightest basic solution conditions.

![Phenolphthalein Structure](image)

Below pH 8.2, colorless

pH 8.2 - 12, pink

The Titrant Concentration – The NaOH titrant solution that you are using has been properly standardized by trained personnel. Standardizing a titrant solution requires established analytical protocols to ensure and exact concentration to 4 significant figures. The NaOH solution was standardized by using anhydrous potassium hydrogen phthalate (KHP), which you will use in a subsequent experiment.
Instructions

1. Clean and rinse a buret in this manner:
   (1) Rinse with a VERY dilute soap-water mixture and rinse with tap water until no soap bubbles are evident.
   (2) Rinse with several milliliters of deionized water from a squirt bottle.
   (3) Rinse twice with ~3 mL of NaOH solution and discard. (This “coats” the inside of the buret with the titrant)

2. Fill the buret with the NaOH solution to approximately the 50.00 mL mark. Do not waste time trying to get exactly 50.00 mL. Attach the buret to the stand, and allow the solution to settle inside for a moment or two.

3. If present, remove the air bubble from the tip of the buret via your lab instructor’s demonstration.

4. Using a clean, dry 250 mL beaker, obtain ~ 100 mL of HCl stock solution and take it to your work area. (This will give you enough for at least 3 titrations without having to go back and forth to the stock solution.)

5. Accurately measure 25.00 mL of your HCl solution by using a transfer pipette, and properly place the solution into a clean* 125 mL flask.

*NOTE: Do not waste your time drying the flask. Just clean it – the flask does not have to be dry! Think about it: The number of moles of H\(^+\) will not change and neither will the volume you accurately measured in the pipette!!

6. Add 2-3 drops of phenolphthalein indicator to the flask and swirl to mix. The solution should remain clear and colorless. DO NOT forget this step. You will have to start over if you do!!

7. Make 110% sure that you added the indicator to the HCl solution. (Now, there’s no excuse: The professor reserves the right to point and laugh at you if you did not ADD THE INDICATOR.)

8. Write down the initial volume of NaOH in the buret on your data sheet using 4 significant figures.

9. Making sure you added the indicator, begin the titration by adding the titrant to the analyte.*

*NOTE: Do not waste time and add one drop at a time during the early stage of the titration. You know that the HCl concentration is ~0.1 \(M\), and you added 25.00 mL. So, go ahead and add ~22 mL of titrant!

10. Making sure the solution is well-mixed, slow down and deliver the titrant drop-wise at this point until you achieve a color change. Create a medium drop rate in the beginning while constantly swirling the solution; then, slow the rate down while keeping a close eye on the solution. It is also very important to incorporate any drops of liquid that are on the inside of the flask, as they may contain H\(^+\) or OH\(^-\) ions! Once you start to see splashes of pink color that persist for a few seconds, add only one drop at a time and maybe even a half-a-drop at a time. **Your GOAL is to obtain the lightest pink color possible.** If the [HCl] is reasonable via the calculations performed in Steps 11-12, keep this flask and match subsequent endpoint colors to the color you achieved here. (This provides good precision.)

11. Write down the final volume of NaOH in the buret in your data table, and use it to calculate the molarity of the HCl solution. It should be very close to the accepted value given by your instructor.

12. Repeat Steps 2–12 until you have 3 molarity values that agree within ±2% of the known HCl concentration and each other.

**NOTE:** To calculate the ±2% value, use the following formula. Yes, your answer may by negative; this means that you’re lower than the accepted value.

\[
\text{% Error} = \left( \frac{\text{Your Value} - \text{Accepted Value}}{\text{Accepted Value}} \right) \times 100
\]
Pre-Lab Exercise

Name: ________________________________

1. Provide the molecular, complete ionic, and net ionic chemical equations for the reaction of HCl and NaOH. Circle the spectator ions in the complete ionic equation.

2. Explain the difference between the terms end point and equivalence point.

3. How many mL of 0.15 M NaOH are required to titrate 25 mL of 0.20 M HCl?

4. A titration required 37.52 mL of 0.1001 M NaOH to reach the end point when titrating 30.00 mL of an HCl solution. What is the molar concentration of the HCl solution based on this datum?
Data & Calculations

Place your experimental data in the following table. You may not need to perform more than 3 trials.

<table>
<thead>
<tr>
<th>Trial #</th>
<th>$V_{initial}$ (mL)</th>
<th>$V_{final}$ (mL)</th>
<th>$\Delta V$ (mL)</th>
<th>Moles of OH$^-$ Required</th>
<th>Volume of HCl Stock (mL)</th>
<th>Calculated [HCl] (mol/L)</th>
<th>$\Delta%$ from Accepted Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>2</td>
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<td>3</td>
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<tr>
<td>6</td>
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<td></td>
</tr>
<tr>
<td>7</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Provide sample calculations here for columns 4–8. Your final molarity of HCl should contain 4 sig figs.
Experiment 16 – The Titration of Soda Ash

Goal: Determine the % purity of an unknown sample of Na$_2$CO$_3$ (soda ash) by titration.

Introduction

A titration is an quantitative analytical method used to determine the unknown amount of a substance (the analyte) by reacting it with a known amount of another substance (the titrant). The chemical reaction between the analyte and titrant must be known, as the stoichiometry between the two can be used to determine how much of the titrant was needed to completely react with the analyte.

A common type of titration is an acid-base titration, where a known amount of an acid (or base) is reacted with an unknown amount of a base (or acid) until a certain pH is reached. By using reaction stoichiometry, the amount of the unknown acid or base can be calculated by knowing how much of the known component was needed to complete the acid/base reaction.

A common way to determine the point at which the titration is complete, or the endpoint, is by using a chemical substance as an indicator. In an acid-base titration, the properties (usually color) of the indicator change with pH, allowing for the endpoint to be determined.

Concept of the Experiment

In this experiment, the amount of soda ash (sodium carbonate, Na$_2$CO$_3$) in a sample will be quantified. This will be determined by titrating a Na$_2$CO$_3$ sample of unknown concentration with a known concentration of HCl. Thus, the Na$_2$CO$_3$ is the analyte and the HCl is the titrant.

There are two parts to the experiment. In Part I, the exact molarity of the HCl solution must be determined by titrating the HCl solution with a pure samples of Na$_2$CO$_3$. This step is called a standardization. It is extremely important that the concentration of the titrant is determined to a high degree of accuracy. In this step, the exact weight of a solid Na$_2$CO$_3$ sample infers the number of moles of Na$_2$CO$_3$ in the flask. Once titrated with the HCl solution, the number of moles of HCl in the volume used to complete the reaction can be determined by using the reaction stoichiometry below. In Part II, the unknown sample of Na$_2$CO$_3$ will be determined with the standardized HCl solution.

The known chemical reaction used in this titration is:

$$2\text{HCl(aq)} + \text{Na}_2\text{CO}_3(s) \rightarrow \text{CO}_2(g) + \text{H}_2\text{O(l)} + 2\text{NaCl(aq)}$$

If the appropriate indicator is used, the endpoint of titration can be assumed to be nearly the same as the equivalence point (You should find out why this is different from the endpoint.) At the equivalence point, stoichiometric equivalent amounts of acid and base have reacted. For the reaction above at the equivalence point, 2 moles of HCl have reacted for every 1 mole of Na$_2$CO$_3$. In Part I of the experiment, use this relationship to calculate the molarity of the HCl solution. In Part II of the experiment, this relationship will again be used to determine the amount of Na$_2$CO$_3$ present in the unknown sample. This is possible because an exact volume of known HCl solution was needed to complete the chemical reaction. Once again, the molarity of HCl will be known from Part I.
Procedure

Obtaining the bulk solids

1. The Na$_2$CO$_3$ (soda ash) samples will be stored in a desiccator in order to keep them dry. Obtain approximately 0.7 g of pure Na$_2$CO$_3$ by weighing the sample using the top-loading balances in the lab and placing it into a 10 mL beaker. Also obtain from the laboratory instructor your unknown, an impure sample of Na$_2$CO$_3$. You will receive approximately 1.5 g of unknown. Record your unknown number on the final results page for this laboratory. Place the entire sample of your unknown in a separate 10 mL beaker. Clearly label both beakers, so that you will know which beaker is the pure Na$_2$CO$_3$ and which is the unknown.

Preparing the HCl solution

2. If a 0.1 M HCl solution has been provided for you by the laboratory instructor, obtain 500 mL of this solution using a 1 L beaker to measure the volume of the solution. We will be determining the molarity of this solution to a greater accuracy.

Weighing the pure Na$_2$CO$_3$(s) samples for titration

3. Three 125 mL Erlenmeyer flasks will be required; label each one. Using the Mettler balances and wax weighing paper, weigh three samples of the pure Na$_2$CO$_3$(s) to the nearest 0.0001 g, each weighing between 0.10 – 0.12 g. Quantitatively transfer each sample into a labelled flask. Record the exact masses of each on the data sheet. To assure quantitative transfer, use distilled water to rinse any particles from the weighing paper into the 125 mL flasks. Add about 15 mL of distilled water to each and swirl gently to dissolve the salt.

Part I: Titrating the pure Na$_2$CO$_3$ (aq) samples to standardize the HCl solution

The pH indicator for the reaction is bromcresol green (0.1% w/w sodium salt of bromocresol green in water). The protonated (acidic) form of bromcresol green indicator is yellow, and the deprotonated (basic) form is blue. At a pH of 3.8 or less, the indicator is in its protonated (green) form. At a pH of 5.5 or higher, the indicator is in its deprotonated (blue) form.

4. Add three drops of bromcresol green indicator to one of the dissolved samples. (The sample should turn blue).
5. Add the HCl solution from the reagent bottle into a buret and record the initial volume. All volumes must be recorded to 0.01 mL.
6. Titrate the blue, basic solution with the HCl solution until a pale green color results. At that point, stop the titration and boil the solution gently for 1 – 2 minutes, taking care that no solution is lost during the process. (Do this by adding distilled water to maintain the volume.) The boiling removes CO$_2$ and hence the weak diprotic acid H$_2$CO$_3$, which causes a poor endpoint. Upon removing the CO$_2$, the solution should turn from pale green back to blue. Cool the solution to room temperature by placing the flask in a 600 mL beaker with an adequate amount of room temperature water. This will help, because the endpoint is more distinctive at room temperature.
7. Wash the inside of the flask's walls with distilled water from a wash bottle, and then continue the titration to the first appearance of yellow that immediately disappears. Add the HCl one-half of a
drop at a time at this point, as the endpoint is extremely close. Titrate until the green color changes to yellow. Record the final HCl volume and calculate the volume that was added.

8. After completing the titration with the first dissolved sample, titrate the other two samples. Do not begin another titration while you are in the middle of a titration! A key to accurate titrations is precision. Strive to acquire a consistent color among all three samples.

9. For each sample of pure Na₂CO₃ titrated with HCl, calculate the molarity of the HCl solution to 4 significant figures.

**Part II : Titrating your unknown soda ash samples**

10. Weigh three separate samples of the dried unknown sample of Na₂CO₃ to the nearest 0.0001 g, each weighing approximately 0.22 – 0.28 g, into separate 125 mL Erlenmeyer flasks. Add about 15 mL of distilled water to each and swirl gently to dissolve the salt. Follow Steps 4 – 8 for titrating the unknown Na₂CO₃.

**Calculation of the percent sodium carbonate in the unknown soda ash sample**

11. Calculate and report the percent of Na₂CO₃ in each sample. The results should be reported to 4 significant figures. Report the average % sodium carbonate on your calculation sheet along with the unknown number!
Experiment 16 – Pre-Laboratory Assignment

Name: ___________________________  Lab Day and Time: _________________________

1. Give the balanced reaction that occurs during the acid-base titration.

2. Why is the solution boiled at the time that it turns green?

3. If 42.15 mL of a 0.1143 M HCl solution is used to titrate 25.08 mL of a Na₂CO₃ solution, what is the molarity of the soda ash solution?
Experiment 16 - Part I: Data and Calculations

Data
For each of your three trials, you must record the mass of pure sodium carbonate used, the initial and final volumes of HCl in the buret, and the calculated volume of HCl that was used.

Calculations
For each trial, calculate the molarity of the HCl solution. Use the average of your three trials for analyzing your unknown in Part II.

Average Molarity of HCl solution (to 4 significant figures): ______________________
**Experiment 16 – Part II: Data and Calculations**

**Data**
For each of your three trials, you must record the mass of unknown sample used, the initial and final volumes of HCl in the buret, and the calculated volume of HCl that was used.

**Calculations**
For each trial, calculate the % (w/w) of Na$_2$CO$_3$. Report the average of your three trials along with your unknown number.

Unknown number = ____________

Percentage of Na$_2$CO$_3$ in the unknown sample = ______________
Experiment 17 – Effectiveness of Commercial Antacids

Goal: Determine the amount of acid neutralized by an antacid.

Introduction

Some medical information is gathered in bizarre ways. This is certainly the case for our early knowledge of stomach chemistry. On June 6, 1822 at Fort Mackinac, Michigan, Alexis St. Martin accidentally shot himself in the stomach while cleaning his shotgun. Dr. William Beaumont stitched the wound. While the edges healed, they did not knit, leaving a hole that was only covered by a flap. The hole led back to the stomach. The contents of St. Martin’s stomach could thus be observed. Dr. Beaumont found that dangling a piece of meat on a string into the stomach caused the excretion of gastric juices, which he removed by the use of a rubber tube. The juices contained approximately $0.1 \ M \ HCl$, among other things [Britannica On-line. Mercyhurst College HAMLET system. Taken from on-line search of “Alexis St. Martin”].

One might think that this strong acid solution would lead to irritation of the stomach wall, since it is capable of dissolving iron. To determine how the stomach is protected, Dr. Horace W. Davenport of the University of Michigan Medical School studied the effects of detergents on dog stomachs. Detergents dissolve lipids (nonpolar fatty substances) and allow them to be washed away. After washing with detergents, the dog stomachs were attacked by the $0.1 \ M \ HCl$ that they naturally contain. Apparently, a healthy stomach is lined with a lipid barrier which prevents attack by ionized acids dissolved in water. Aqueous solutions are polar, and would not penetrate a nonpolar stomach lining.

Weak acids, like aspirin (acetylsalicylic acid), can attack the stomach wall because they are undissociated in water, and the undissociated form may be nonpolar enough to penetrate the lipid barrier. After the acid penetrates the stomach lining, it finds itself in a pH neutral environment where it dissociates to a much greater extent than it does in the acid environment of the stomach. The acetylsalicylate ion is proteolytic (protein-degrading) and destroys some of the protein of the stomach wall. This ultimately leads to the loss of 1-2 mL of blood when the average person takes two aspirin. Some foods apparently can either overstimulate acid production in the stomach or reduce the stomach wall’s resistance to the acid, and we experience “acid indigestion.”

Various “cures” for “acid indigestion” are available, including the following:

Tums® contain 500 mg of calcium carbonate, CaCO$_3$. Calcium carbonate is only very slightly soluble in water. The dissociation is given by:

$$\text{(Rxn 1) \quad \text{CaCO}_3(s) \rightleftharpoons \text{Ca}^{2+}(aq) + \text{CO}_3^{2-}(aq)}$$

so the concentration of the base, CO$_3^{2-}$, is present in low concentrations. In the presence of an acidic environment, such as that found in the stomach, a far greater amount of CaCO$_3$ dissolves than in a pH neutral environment. This is because the CO$_3^{2-}$ ion couples with protons (H$^+(aq)$) in the acidic environment, forming bicarbonate ion, HCO$_3^-$. As predicted by LeChatelier’s Principle, as the CO$_3^{2-}$ ion of Rxn 1 is removed by the formation of HCO$_3^-$ in Rxn 2, the equilibrium of Rxn 1 will shift to the right, to compensate for the loss of CO$_3^{2-}$. Thus, more CaCO$_3$ dissolves.
The equations for the removal of the carbonate ion, bicarbonate ion, and carbonic acid are:

(Rxn 2) \[ \text{H}^+(aq) + \text{CO}_3^{2-} (aq) \rightleftharpoons \text{HCO}_3^-(aq) \]

(Rxn 3) \[ \text{H}^+(aq) + \text{HCO}_3^- (aq) \rightleftharpoons \text{H}_2\text{CO}_3(aq) \]

(Rxn 4) \[ \text{H}_2\text{CO}_3(aq) \rightleftharpoons \text{CO}_2(g) + \text{H}_2\text{O}(l) \]

Rxn 2 shows the carbonate ion acting as a base to neutralize H\(^+\). Rxn 3 and Rxn 4 show that during the neutralization process, there are significant quantities of HCO\(_3^-\) and H\(_2\)CO\(_3\) present. (Actually, the H\(_2\)CO\(_3\) is present largely in the dissociated form of CO\(_2\) and H\(_2\)O.) Since a mixture of a weak acid and its conjugate base is a buffer, this system will contain both the HCO\(_3^-\)/CO\(_3^{2-}\) and H\(_2\)CO\(_3\)/HCO\(_3^-\) buffers during various stages of the neutralization.

If HCl is added to the H\(_2\)CO\(_3\)/HCO\(_3^-\) buffer, the acid is neutralized as HCO\(_3^-\) (bicarbonate ion) accepts the proton of the acid and converts into H\(_2\)CO\(_3\) (carbonic acid). The pH will change very little during this process. This is good for stabilizing your stomach, but it leads to difficulties when we try to determine the strength of an antacid by titration using an acid. A distinct indicator “endpoint,” or color change, requires a rapid change of pH near the equivalence point of the titration, and this does not occur in a buffered solution.

Rolaids\textsuperscript{®} contain 300 mg of dihydroxyaluminum sodium carbonate, NaAl(OH)\(_2\)CO\(_3\). Rolaids are said to consume “47 times their weight” in excess stomach acid. The dihydroxyaluminum compound contains two ions that can serve as bases: carbonate and hydroxide. The carbonate ions of Rolaids neutralize acid according to Rxn 2 and Rxn 3 above. The hydroxide ions of Rolaids neutralize acid by Rxn 5:

(Rxn 5) \[ \text{OH}^-(aq) + \text{H}^+(aq) \rightleftharpoons \text{H}_2\text{O}(l) \]

Milk of Magnesia is an aqueous suspension of the rather water-insoluble compound magnesium hydroxide, Mg(OH)\(_2\). It would be impossible for a human to consume a concentrated solution of aqueous hydroxide ions. However, Mg(OH)\(_2\) is a slightly insoluble compound, as seen in Rxn 6. The low concentration of hydroxide ion in solution for a Mg(OH)\(_2\) slurry greatly lessens the effects of the strong OH\(^-\) base on tissue.

(Rxn 6) \[ \text{Mg(OH)}_2(s) \rightleftharpoons \text{Mg}^{2+}(aq) + 2\text{OH}^-(aq) \]

\( K_{sp} \) of Mg(OH)\(_2\) = 1.2\times10^{-11} \text{ (at 18 } ^\circ \text{C)} \]

The concentration of hydroxide ion is never high enough to damage body tissue. As the hydroxide ion in solution reacts with stomach as (as given in Rxn 5), more Mg(OH)\(_2\) dissolves, as described in Rxn 6. This provides additional OH\(^-\) until the Mg(OH)\(_2\) is fully dissolved.

The old standby, sodium bicarbonate (NaHCO\(_3\)), has fallen in popularity because of “anti-sodium advertising”, although it is still an ingredient (465 mg) in Alka-Seltzer\textsuperscript{®}. Alka-Seltzer also contains CaCO\(_3\) (280 mg), KHCO\(_3\) (300 mg), and citric acid (900 mg, formula C\(_6\)H\(_8\)O\(_7\)). It seems odd for an “antacid” to actually contain an acid, but citric acid is so weak that it is undissociated in the stomach where high H\(_3\)O\(^+\) concentrations from the HCl force the citric acid to remain in protonated form. The citric acid does dissociate enough in pH neutral water enough to generate enough H\(^+\) to react with the CO\(_3^{2-}\) and HCO\(_3^-\) ion present to produce CO\(_2\) by Rxn 2 and Rxn 3. This reaction causes the bubbling that you observe when you dissolve an Alka-Seltzer tablet in water.
The citric acid dissociation equilibrium involving citric acid is:

\[
(Rxn \ 7) \quad \text{HOC(CH}_2\text{COOH)}_2\text{COOH(aq)} \rightleftharpoons H^+(aq) + \text{HOC(CH}_2\text{COOH})\text{COO}^-(aq)
\]

\[K_a = 7.1 \times 10^{-4} \quad \text{(at 18 °C)}\]

Some generic antacids (for example CVS® brand) may contain mixtures of Mg(OH)_2 and Al(OH)_3, typically 200 mg each. Since these compounds are insoluble, one way to write equations for the reactions with acids is

\[
(Rxn \ 8) \quad \text{Mg(OH)}_2(s) + 2H^+(aq) \rightleftharpoons 2\text{H}_2\text{O(l)} + \text{Mg}^{2+}(aq)
\]

\[
(Rxn \ 9) \quad \text{Al(OH)}_3(s) + 3H^+(aq) \rightleftharpoons 3\text{H}_2\text{O(l)} + \text{Al}^{3+}(aq)
\]

Rxn 8 is an equilibrium that can be generated by combination of Rxn 5 and Rxn 6. The equilibrium constant for Rxn 8 would therefore be the product of the equilibrium constants associated with the equilibria of Rxns 5 and 6.

**Notes on Calculations and Procedure**

To determine the acid-neutralizing power of an antacid, it would seem logical to simply titrate the tablet with acid to see how much will be consumed in a reaction such as:

\[
(Rxn \ 10) \quad \text{CaCO}_3 + 2\text{HCl(aq)} \rightarrow \text{H}_2\text{CO}_3(aq) + \text{CaCl}_2(aq)
\]

In a titration, however, we depend on a rapid change of pH at the equivalence point as very small amounts of titrant are added. Only then can the endpoint be easily detected. Because antacid tablet solutions will often produce a buffered solution during a titration, rapid pH changes are resisted, and endpoints are less distinct.

In this experiment, we will reduce this problem by adding an excess of HCl of known concentration to the antacid tablet, and boil the solution. The excess HCl will protonate the carbonate and bicarbonate ions in solution producing H_2CO_3, which converts into gaseous CO_2. By boiling the solution, we reduce the solubility of gas in the solution, thereby expelling the CO_2. This forces Rxns 2, 3 and 4 to go to completion, removing the buffer solution and leaving only H^+, which can be back-titrated with standard NaOH.

If we know the number of moles of HCl \((n_H)\) that were initially added to the antacid solution, and we know the number of moles of NaOH \((n_{OH})\) that were added to neutralize the acid, then we can calculate the number of moles of acid that were neutralized by the antacid tablet \((n_{ant})\) by difference:

\[
\begin{align*}
    n_{ant} + n_{OH} &= n_H \\
    n_{ant} &= n_H - n_{OH}
\end{align*}
\]
Let’s do a sample calculation. Suppose a 0.80 g antacid tablet contains 200 mg of NaHCO₃ (FW = 84 g/mol). You treat this with 50.00 mL of 0.100 M HCl, and boil the solution. This completely dissolves the antacid, and presumably removes all carbonate species, leaving only non-neutralized H⁺ in the solution. You then back-titrate with 26.20 mL of 0.100 M NaOH solution to the phenolphthalein endpoint (faint pink color). The steps involved in calculations are given below.

Steps to the sample calculation

1. Determine the number of moles of HCl added to the antacid.
   \[ n_{H} = (0.100 \text{ mole H}^+/\text{L})(0.0500 \text{ L}) = 0.00500 \text{ mol HCl} \]

2. Determine the number of moles of NaOH used in the back-titration.
   \[ n_{OH} = (0.100 \text{ mole OH}^-/\text{L})(0.0260 \text{ L}) = 0.00260 \text{ mol NaOH} \]

3. Recognize that for every mole of NaOH added, one mole of H⁺ is neutralized. So of the 0.00500 mol HCl initially added to the flask, 0.00260 mol of HCl is neutralized by the NaOH added. That means that 0.00238 mol of HCl was neutralized by the antacid tablet. This is given by:
   \[ n_{\text{ant}} = n_{H} - n_{OH} \]
   \[ n_{\text{ant}} = 0.00500 \text{ mol} - 0.00260 \text{ mol} = 0.00238 \text{ mol} \]

   Recognize that \( n_{\text{ant}} \) is not necessarily the number of moles of antacid present. It is the number of moles of HCl that was neutralized by the antacid tablet.

   You were told that the antacid present in you tablet was the bicarbonate ion, HCO₃⁻. This ion reacts with H⁺ in a 1:1 stoichiometric ratio as given by Rxn 3. So in this case, the 0.00238 mol of H⁺ that was neutralized by the bicarbonate tells us that 0.00238 mol of bicarbonate ion must have been present in your tablet. This says that there was 0.00238 mol of NaHCO₃ present in the tablet. Therefore, since the formula weight of NaHCO₃ is 84.00 g/mol, you can verify the mass of NaHCO₃ present by:
   \[ \text{mass of NaHCO}_3 \text{ present} = (0.00238 \text{ mol})(84.00 \text{ g/mol}) = 0.200 \text{ g} \]

   In rare cases where the antacid tablet contains buffers other than the carbonate system, the titration may produce an indistinct endpoint if treated as described in this experiment.
Concept of the Experiment

*Standardization of the NaOH Solution*

Three or four experimenters in the class will perform this portion of the experiment. Their results will be shared with the other members of the class for everyone’s use.

These students will determine the concentration of (or “standardize”) the class’ NaOH solution by titration with potassium hydrogen phthalate (KHC₈H₄O₄, nicknamed KHP, FW = 204.22 g/mol) by the 1:1 reaction

\[
\text{OH}^- + \text{KHC}_8\text{H}_4\text{O}_4 \rightarrow \text{H}_2\text{O} + \text{KC}_8\text{H}_4\text{O}_4^- 
\]

KHP is a **primary standard**. This means that it can be obtained and maintained in a highly pure form, which can be easily and directly weighed. The weighed sample is dissolved in water and titrated with sodium hydroxide solution to determine the exact concentration of the solution. For example, if 0.302 g of KHP required 15.0 mL of an NaOH solution, the molarity of the NaOH solution could be calculated as follows:

\[
\begin{align*}
\text{moles of KHP} &= \frac{0.302 \text{ g}}{204.22 \text{ g/mol}} = 0.00148 \text{ moles KHP} \\
0.00148 \text{ moles of KHP} &= 0.00148 \text{ moles NaOH} \\
M \text{ NaOH solution} &= \frac{0.00148 \text{ moles NaOH}}{0.0150 \text{ L}} = 0.0986 M \text{ NaOH}
\end{align*}
\]

**Evaluation of Antacids**

The students that did not perform the standardization of the NaOH solution or the HCl solution will perform this portion of the experiment.

Although the effectiveness of antacid tablets may be related to factors other than their acid neutralizing ability, the goal of this experiment is to calculate the effectiveness of an antacid for neutralizing HCl. You will evaluate the antacids in several ways:

1. moles HCl neutralized per dose (tablet);
2. moles HCl neutralized per gram of tablet;
3. moles HCl neutralized per dollar cost per dose.
Experiment 17 – Pre-Laboratory Assignment

Name: _______________________________  Lab day and Time: ___________

1. If 0.200 g of KHP (F.W. = 204.22 g/mol) require 22.30 mL of sodium hydroxide solution in a titration what is the molar concentration of the sodium hydroxide solution? (Hint: use Rxn 11)

2. If we write the overall equation for the reaction of Tums® with stomach acid as follows:

   \[ \text{CaCO}_3(s) + 2\text{HCl}(aq) \rightarrow \text{H}_2\text{CO}_3(aq) + \text{CaCl}_2(aq) \]

   (a) Suppose each tablet contains 500 mg of CaCO₃. Convert this value to moles of CaCO₃.

   (b) Calculate the number of moles of HCl required to react with each tablet (Hint: refer to Rxn 10)

   (c) Calculate the volume of 0.100 M HCl that is required to react with one tablet (500 mg CaCO₃)

   (d) The density of 0.100 M HCl is 1.003 g/mL. Find the mass of the volume of 0.100 M HCl that you calculated in part (c).

   (e) Assume that a Tums® tablet has a mass of 2.0 g. (The calcium carbonate content is 500 mg, or 0.50 g). Given your answer to part (d), does Tums® consume at least “47 times its weight (2.0 g) in stomach acid” as has been claimed?
Procedure

Part 1 – Standardization of NaOH Solution

(May be done by a select group of students in place of antacid analysis. Consult your lab instructor)

1. Rinse a buret with a few mLs of distilled water and then a few mLs of NaOH. Then fill the buret with the NaOH solution.

2. Label two 125 mL Erlenmeyer flasks as Trial 1 and Trial 2. Take the flasks into the balance room. Place a piece of analytical weighing paper onto the balance. Tare the paper. Place about 0.20 g of KHP into the first Erlenmeyer flask. You must record its mass to 4 decimal places using the analytical balances. Repeat for the second flask.

3. Add 15-25 mL of distilled water to the KHP to dissolve it, then add 2-3 drops of phenolphthalein indicator, and titrate the solution with the sodium hydroxide solution. Rinse the inside walls of the Erlenmeyer flask when you approach the endpoint. Repeat for the second trial. If the two trials do not agree, you should perform additional trials. Calculate your average value for the molarity of the NaOH solution from your two good titrations.

4. Place your results on the blackboard. When all NaOH standardization results are on the board, determine the overall average molarity of the NaOH solution. This result must be shared with the other students for their calculations regarding the analysis of the antacids.
Procedure

Part 2 – Analysis of Antacid Tablet

1. Obtain an antacid tablet from one of the bottles supplied. Using an analytical balance and weighing paper, record the mass of the tablet to 4 decimal places using the analytical balance. If your sample is a powder, record the mass of one dose.

2. Take the tablet back to your drawer. Using your spatula, cut the about in half. Label two 125 mL Erlenmeyer flasks as Trial 1 and Trial 2. Take the two halves and the two flasks back to the weighing room and get the masses of each half-tablet. If a powder is supplied, weigh about 0.5 g precisely. After recording the masses for each trial, place the samples in the appropriate flasks.

3. Record the brand, price, number of tablets in a full container, ingredients, and mass of each ingredient.

4. Use the flask labeled Trial 1. Add 25 mL of distilled water. It is not necessary to completely dissolve the tablet before the next step.

5. Rinse and treat the buret with standard HCl (the molarity should be written on the bottle). Deliver 48.00 mL to the Erlenmeyer flask and sample. Record the exact amount you delivered.

6. Heat the mixture to a gentle boil. Allow it to gently boil for 5 minutes on a hotplate or wire gauze over a Bunsen burner.

7. Remove the flask from the heat, using tongs. Allow the flask to cool for a couple minutes by running room-temperature water over the flask. Do not break the flask by running cold water over hot glass!

8. Add three drops of phenolphthalein indicator.

9. If the color of the solution is not pink at this point, go to step 10. If the addition of the phenolphthalein indicator turns the solution pink, that means that you did not add enough acid to the solution to neutralize all of the antacid. The pink color indicates a basic solution. Add standard HCl solution in measured quantities using your buret, until the color disappears. Boil again for five more minutes. Cool as before, and add one additional drop of phenolphthalein indicator. Go to step 10.

10. Treat and fill a buret with standard NaOH solution. Back titrate the excess HCl. Rinse down the inside walls of the flask with distilled water as you near the endpoint. Titrate to the first permanent (20-30 s) color of faint pink.

11. Repeat the titration (steps 3-10) for your second trial. If your results do not agree, you must perform additional trials.
Experiment 17 - Data and Calculations

Standardization of NaOH Solution (If this part has been performed)

Show all data and calculations necessary to determine the molarity of the NaOH solution that is used. You should have performed at least 2 trials. The data should include:

(i) Mass of KHP
(ii) Moles of KHP
(iii) Initial and Final Buret Readings
(iv) Volume of NaOH used
Experiment 17 - Data

Analysis of Antacid Tablet

Show all data for the analysis of your antacid. You should have performed at least 2 trials. The data should include:

(i) Brand and ingredients of tablet.
(ii) Mass of entire tablet.
(iii) Mass of antacid sample tested.
(iv) Initial and final buret readings for HCl addition.
(v) Volume and molarity of HCl added.
(vi) Initial and final buret readings for NaOH titration.
(vii) Volume and molarity of NaOH used
Experiment 17 – Calculations

Analysis of Antacid Tablet

Show all calculations and results in your analysis of the antacid tablet. These include the calculation of the following:

(i) Moles of HCl added.
(ii) Moles of NaOH added in the titration.
(iii) Moles of HCl consumed by antacid sample.
(iv) Moles of HCl consumed per gram of antacid.
(v) Moles of HCl consumed per antacid tablet.
Experiment 17 – Post-lab Questions

1. After comparison with classmates data, what antacid neutralized the most acid per gram?

2. What was the active ingredient in the answer to question #1?

3. Give a clear, concise definition of back-titration.