# CHE118 Experiments

# Fall 2024
# Spring 2025
# Summer 2025

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## Experiment 21: Calibration Curve Review

### Purpose

The purpose of this experiment is to reintroduce students with working in a general chemistry lab and to review the basics of making and using a calibration curve.

### Background

This absorbance of light will follow the Beer-Lambert Law (Beer's Law), which states that the absorbance of light by a solute in solution is directly proportional to its concentration. The mathematical equation is A = abc, where A is absorbance, a is the molar absorptivity coefficient, b is the path length, and c is concentration.

Calibration curves are graphs that show the relationship between concentration and absorbance of light by the solute. Absorbance is dependent on the concentration of solute in the solution. Therefore, concentration is on the x-axis and absorbance is on the y-axis. The data points obtained in lab are plotted using Excel or Google Sheets. The computer program will put a best-fit line through the data points. The equation of the linear line is obtained, and this equation shows the mathematical relationship in the

$y = mx + b$ format which translates to $A = mC + b$ *(this b is not path length)*

*The A is absorbance, m is the slope of the line, C is the concentration, and b is the y-intercept.*

The spectrophotometers that are in the general chemistry laboratory cover the wavelength range of visible light. Therefore, in order to measure the absorbance of light by the solute, the solute must produce a colored solution.

The **first step** to prepare a calibration curve is to make the standard solutions. These are solutions of known concentration. The **second step** is to determine the best wavelength at which to measure the absorbance. The best wavelength is typically the wavelength at which the solute absorbs the maximum amount of light, which is referred to as $\lambda_{max}$. The **third step** measures the absorbance of light by the standard solutions and sample solutions, using the spectrophotometer set to $\lambda_{max}$. The **fourth step** is to plot the data to make a graph.

### Chemicals

Blue Dye # 1 stock solution (2.0 %wt)
Blue Dye # 1 sample solution of unknown concentration
Deionized water

### Equipment

50 mL volumetric flasks
10 mL graduated pipet and pipettor
Parafilm
Plastic droppers
Test tubes, test tube rack
Spectrophotometer

## Procedure

**Part A: Preparing Standard Solutions** *(Solutions of known concentrations.)*
Use the provided stock solution to prepare the standard solutions. Calculate the needed volume of the stock solution for each standard solution, using the dilution equation: $M_c \cdot V_c = M_d \cdot V_d$ *(you are solving for Vc)*

The concentrations of the standard solutions should be:

| Concentration of Standard Solution, %wt | Vc, Volume of Stock Solution to pipet into 50 mL volumetric flask |
|---|---|
| 0.10 | |
| 0.20 | |
| 0.40 | |
| 0.60 | |

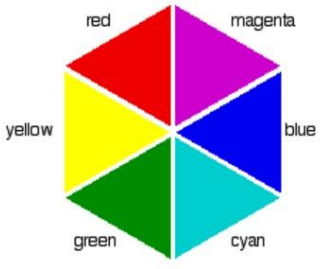
1) Use the graduated pipet to deliver the correct amount of stock solution to each 50 mL volumetric flask.
2) Dilute to the calibration mark with deionized water. Make sure the bottom of the meniscus sits exactly at the calibration mark on the neck of each flask.
3) Cover the flask with Parafilm, and invert 50 times to mix.

**Part B: Determining the $\lambda_{max}$** *(finding the best wavelength for absorbance of light by the dye)*

1) Use the 0.60% standard solution for this part of the experiment. Place this solution into a small test tube, about ¾ full. Put DI water into another test tube, also about ¾ full. The DI water is your blank that you will use to zero the spectrophotometer.
2) The dye looks blue because it absorbs light in the 560 nm – 650 nm region of visible light. Refer to Figure 1. You need to determine the best wavelength in this region at which the dye absorbs light. Turn on the spectrophotometer set the software to the wavelength scan option. Scan from 400 nm to 700 nm.

| color region | wavelength (nm) |
|---|---|
| violet | 380 - 435 |
| blue | 435 - 500 |
| cyan | 500 - 520 |
| green | 520 - 565 |
| yellow | 565 - 590 |
| orange | 590 - 625 |
| red | 625 - 740 |

Figure 1

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3) Put the test tube with the DI water into the sample compartment of the spectrophotometer, and close the lid. Press the zero button. This sets the zero point on the spectrophotometer and subtracts out any absorbance of light by the glass and DI water.
4) Put the test tube of standard solution into the sample compartment and close the lid. Run the wavelength scan, which will provide a graph of wavelength vs. absorbance. Record the wavelength that yields the maximum absorbance.

**Part C: Measure the absorbance of light for each solution (standards and sample)**

1) Make sure the spectrophotometer is set to $\lambda_{max}$ for the wavelength.
2) Use the DI water as the blank to zero the spectrophotometer.
3) Transfer each standard solution and unknown into its own test tube, about ¾ full.
4) Measure the absorbance of light for each solution, and record these values into your notebook.

**Part D: Prepare a calibration curve and Determine the concentration of the unknown**

Follow the instructions in the Calibration Curve video and use Excel or Google Sheets to prepare a calibration curve. The concentration is the independent variable, so that is on the x-axis. Absorbance is the dependent value, so that is on the y-axis. Plot a scatter plot, linear, and put the best-fit line on the graph. Display the equation of the line on the graph, along with the $R^2$ value. The $R^2$ value is not part of the equation of the line, but instead is an indication of how well the data points fit on the line. In other words, how well did you prepare your standard solutions? ($R^2$ should be 1.000 and the b value should be 0.000 .)

Use the equation of the line to solve for the unknown concentration. The equation of the line is in the y=mx+b format. Y is the absorbance and X is the concentration. You know the absorbance of the unknown solution, so solve for X. Show this calculation in your notebook, and report the concentration of the unknown with correct units.

**Print your calibration curve graph and attach it in your notebook.**

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## Experiment 22: Spectrophotometric Determination of Iron in a Vitamin

*(This experiment is by E. L. Pool and D. Copeland (with edits by R. Sandwick and M. J. Simpson), Middlebury College)*

### Purpose

You will use a spectrophotometer to determine the amount of iron in a multivitamin to see if the manufacturer's claim is correct.

### Background

Iron itself is not a huge absorber of light, but when $Fe^{2+}$ binds to phenanthroline, it forms a highly stable red/orange-colored species. By quantifying the color with a spectrophotometer, we can deduce the concentration of iron in the diluted sample solution and back-calculate the amount of iron in the original vitamin. This value can then be compared to the manufacturer's claim on the bottle.

The formation of the red/orange-colored iron-phenanthroline complex requires the iron to be in the $Fe^{2+}$ form, and the procedure thus includes the reagent hydroxylamine hydrochloride that will reduce all iron in the sample to the $Fe^{2+}$ form. Ammonium acetate is used to control the pH of the solution since this also affects the absorbance. The red/orange iron-phenanthroline complex absorbs light at 510 nm. To quantify the intensity of the color, you can use the spectrophotometer to measure the absorbance.

Iron standard solutions will be prepared and their absorbance of the 510 nm light measured. The known concentrations and absorbance values will be used to generate a calibration curve. The equation of the best-fit line on the calibration curve will be used to quantitate the iron in the known quality control solution and vitamin solution.

### Chemicals

| | |
|---|---|
| 1.0 M ammonium acetate | 1% hydroxylamine hydrochloride |
| 0.30% o-phenanthroline | 0.025 mg/mL $FeSO_4$ stock solution |
| 1.0 M hydrochloric acid | Ferrous Ammonium Sulfate *(for QC check)* |
| Iron Vitamin tablet | $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ FW = 392.16 |

### Equipment

| | | |
|---|---|---|
| Pipets, (4) 10 mL, (2) 1 mL | Beakers (5), 50 mL and 100 mL | Parafilm |
| 50 mL vol. flask (4) | Thermometer | Hotplate (use in the hood) |
| Test tubes, large | Test tubes, small | Spectrophotometer |
| Test tube rack | Mortar & Pestle | Graduated cylinder,10 mL |
| Filter paper | Balance, weigh paper | |

**Procedure**

There are three parts to this experiment procedure:

A) The vitamin sample will be prepared for analysis. The solid sample must be dissolved, filtered, diluted, and chemically adjusted to produce a colored solution.

B) The known iron sample will be prepared for analysis as a way to check on the accuracy of the experiment (quality control). The solid sample must be dissolved, diluted, and chemically adjusted to produce a colored solution.

C) The standard iron solutions must be made from the stock solution and chemically adjusted to produce colored solutions.

**Part A: Vitamin Sample Preparation**

The vitamin sample preparation requires boiling the crushed iron vitamin in acid to release the iron, and filtering the solution to remove particulates that could interfere in the spectrophotometer measurements.

1) Obtain one vitamin tablet. Use the lab balance to find the mass of the tablet. Place the tablet on a tared piece of weighing paper.
2) Crush the tablet with a mortar and pestle until it is a fine powder.
3) Measure 0.050 g – 0.055 g of the vitamin powder onto a new piece of tared weighing paper. Record the exact mass and then transfer into a 100 mL beaker.
4) Add 15 mL of 1.0 M HCl solution to the beaker. Swirl to mix.
5) Heat the mixture on a hotplate in the fume hood. Swirl often. Do not leave your sample unattended. Use a thermometer to keep your sample at 70 – 80°C for approximately 10 minutes.
6) Bring your sample back to your lab bench and filter the sample directly into a 50 mL volumetric flask. Rinse the beaker with DI water several times to be sure to transfer all of the sample solution to the filter.
7) Dilute the sample to the graduation mark on the 50 mL flask, using DI water. Cover the flask with Parafilm and invert 50 times to mix.
8) Pipet **2.0 mL** of this solution into a new 50 mL volumetric flask. Dilute to the graduation mark with DI water. Cover with Parafilm and invert 50 times to mix. ***This solution will be the vitamin solution you will use in Part C.***

**Part B: Known Iron Sample Preparation**

The known iron sample requires the dissolving of the solid sample. Heating and filtering are not required.

1) Measure 0.080 g of the known iron sample onto a new piece of tared weighing paper. Transfer this mass into a 50 mL volumetric flask.
2) Add 15 mL of 1.0 M HCl solution to the volumetric flask. Swirl to mix and dissolve.
3) Dilute the sample to the 50 mL graduation mark on the flask, using DI water. Cover the flask with Parafilm and invert 50 times to mix.
4) Pipet **1.0 mL** of this solution into another 50 mL volumetric flask, and dilute to the graduation mark with DI water. Cover with Parafilm and invert 50 times to mix. ***This is the known iron sample solution that you will use in Part C.***

**Part C: Standard Solutions & Color Preparation for all Solutions**

1. Label 8 large test tubes as blank, numbers 1 – 5, Known Fe, and Vitamin. Work with all 8 test tubes at the same time for the following steps.

2. Label 5 small beakers with the names of the stock solutions: ammonium acetate, hydroxylamine HCl, o-phenanthroline, iron stock solution, and DI water. Fill these beakers with a slight excess of the needed volumes.

3. To each of the eight test tubes add 1 mL of each: ammonium acetate, hydroxylamine HCl, and o-phenanthroline.

4. To test tubes 1 – 5, add the volumes of iron stock solution and DI water indicated in Table 1. Record the volumes used in your notebook.

5. To the test tube labeled "Blank", do not add any iron standard. Add the DI water indicated in Table 1.

6. To the test tube labeled Known Fe, use 5.0 mL of the diluted Known Fe solution prepared in step 4 of the **Known Iron Sample Preparation** directions. Add 2.0 mL of DI water as indicated in Table 1. (Do not add iron stock solution.)

7. To the test tube labeled Vitamin, use 5.0 mL of the diluted vitamin solution prepared in step 8 of the **Vitamin Sample Preparation** directions. Add 2.0 mL of DI water as indicated in Table 1. (Do not add iron stock solution.)

6. Cover the test tubes with Parafilm and invert several times to mix well. Let the test tubes sit for 10 minutes.

**Table 1.** *(volumes are in mL)*

| **Standard** | **Blank** | **1** | **2** | **3** | **4** | **5** | **Known Sample** | **Vitamin** |
|---|---|---|---|---|---|---|---|---|
| Vol. ammonium acetate | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| Vol. hydroxyam HCl | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| Vol. o-Phen | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 | 1.00 |
| Vol. Fe stock added (0.025 mg/mL) | 0 | 0.30 | 0.50 | 1.0 | 1.5 | 2.0 | Vol Known Fe soln 5.0 | Vol Vitamin soln 5.0 |
| Vol. DI $H_2O$ | 7.0 | 6.7 | 6.5 | 6.0 | 5.5 | 5.0 | 2.0 | 2.0 |
| Total vol. in test tube | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 | 10.00 |
| Conc. Fe mg/L In the standard solutions | 0 | Calculate using the dilution equation: Mc * Vc = Md * Vd, solve for Md | | | | | ???? | ???? |

## Spectrophotometric Analysis

1. Get 8 small test tubes. Label each small test tube, and transfer some of each solution from its large test tube to its corresponding small test tube.

2. Set the wavelength on the spectrophotometer to 510 nm. Use the blank solution to zero the spectrophotometer.

3. Measure the absorbance of each solution in the remaining test tubes. Record the absorbances in your notebook.

**Calculations and Results**

The absorbance measurements of the standard solutions are used to prepare a calibration curve. The equation of the best-fit line of the calibration curve is used to determine the concentration of iron in the diluted Vitamin solution and the diluted Known Fe solution. These results are then used to determine the amount of iron present in the original vitamin and the original Fe sample. (Do not graph the Known Fe QC and Vitamin sample.)

Print your graph, with the equation of the line and $R^2$ on the graph.

Follow these steps for your calculations of Fe in the Vitamin:

1. The concentration units for the calibration curve were mg/mL. So, the concentration of iron in the Vitamin solution is in units of mg/mL. You prepared 50 mL of the diluted solution, by using 2.0 mL of the digested solution. Reverse the sample preparation math to calculated the mg Fe in the sample-prep beaker.

**(mg/mL Fe from best-fit eqn.) (10 mL / 5 mL) (50 mL / 2.0 mL) (50 mL) = mg Fe**

2. Before you can compare this value of mg Fe to the amount listed on the bottle, you have to consider that not all of the crushed pill was used to make the first solution. The mg of Fe determined are the mg in the crushed sample you put in the beaker.

**(mg Fe) / (grams in beaker) = mg Fe / g used**

3. Now you can calculate the mg of Fe in a whole tablet, which is how the Fe content is displayed on the bottle's label.

**(mg Fe / g used) x (g of whole pill) = mg Fe in a whole pill**

4. Calculate the % error for your result. This will give information as to how your results match up to the bottle label, rather than saying 'the results seem good' when you write your conclusion. A % error less than 5% is good.

$$\% \text{ Error} = \frac{/ \text{ mass Fe label} - \text{mass Fe exp.} /}{\text{mass Fe label}} \times 100$$

Follow these steps for your calculations of Fe in the Known Iron Sample:

1. The concentration units for the calibration curve were mg/mL. So, the concentration of iron in the Known Iron solution is in units of mg/mL. Reverse the sample preparation math to calculated the mg Fe in the original known sample used to make the diluted solution that was put into the spectrophotometer.

**(mg/mL Fe from best-fit eqn.) (10mL / 5mL) (50mL / 1mL) (50mL) = mg Fe in original sample used**

2. Once you know the mg of Fe in the original sample used, calculate the %Fe as follows:

***% Fe = (mg of Fe in original sample used) / (mg of original known sample used) x 100***

3. Calculate the % error for your result. This will give information as to how your results match up to the known value, rather than saying 'the results seem good' when you write your conclusion. A % error of less than 5% is good.

How do you know what the true value is? Calculate it.

%Fe True Value = ( (55.845 g/mole Fe) / (392.14 g/mole FW ) ) x 100 =

$$\% \text{ Error} = \frac{/ \quad \% \text{ Fe True Value} - \% \text{ Fe exp.} \quad /}{\% \text{ Fe True Value}} \times 100$$

## Experiment 23: The Kinetics of the Crystal Violet and Sodium Hydroxide Reaction

**Purpose** To study the kinetics of the reaction between crystal violet (CV) and sodium hydroxide (NaOH). This will involve the determination of the rate law equation; finding the values of the exponents and the value of the rate constant.

### Background

The SDS for crystal violet has several warnings; health hazard, exclamation mark, and environmental hazard are some of them. However, crystal violet has been used for several commercial purposes; bacterial and fungal infection-treatment creams, as well as dye for wood, silk, and paper *(ref. PubChem, Gentian Violet, accessed 4/16/2021)*.

This crystal violet compound has an intense, violet color, which makes it ideal for study with a spectrophotometer. The CV compound consists of the CV cation and chloride anion. When a solution of crystal violet is mixed with a solution of hydroxide ion, a reaction occurs in which the product formed is colorless:

$$CV^{1+}_{(aq)} + OH^{1-}_{(aq)} \rightarrow CVOH_{(aq)}$$

*violet* *Colorless* *Colorless*

The CV solutions you will use for this kinetics experiment will start as violet colored solutions, and when mixed with the sodium hydroxide solution, will fade to colorless. You will use the spectrophotometer to record the absorbance of 590 nm light by the $CV^{1+}$ ion for several minutes, for several trials. The absorbance values will decrease, since the concentration of $CV^{1+}$ decreases over time (reactants get used).

The rate law equation has a general format of reaction rate = $k\,[A]^m\,[B]^n$
For this experiment, the more specific format is reaction rate = $k\,[CV^{1+}]^m\,[OH^{1-}]^n$

To determine the exponents in the rate law equation, several trials of the reaction will be done. The experiment is set up so we can study how the change in concentration of only one reactant affects the rate of the reaction. Therefore, one reactant's initial concentration is different for two of the trials, while the other reactant's initial concentration is kept the same.

When the initial concentration of the hydroxide ion is the same for two trials, and the $CV^{1+}$ initial concentration is different for those two trials, this allows for the determination of the $CV^{1+}$ exponent.
When the initial concentration of $CV^{1+}$ is the same for two trials, and the $OH^{1-}$ initial concentration is different for those two trials, this allows for the determination of the $OH^{1-}$ exponent. *(Note: The concentration of hydroxide is much more than CV, which may influence the calculated hydroxide exponent.)*
The general format for the mathematical equation for the calculation of the exponent is:

**(ratio of initial concentrations)$^m$ = (ratio of reaction rates)**

Be sure to ratio the appropriate two trials for each reactant to solve for the exponents.

**Chemicals**
0.10 M NaOH solution ***(You will only need approximately 20 mL of NaOH solution.)***
2.0 x $10^{-5}$ M CV solution ***(You will only need approximately 20 mL of CV solution.)***

**Equipment**
Test tubes Test tube rack Spectrophotometer
5 mL pipets (three), pipettor
Parafilm squares, timer, three beakers (for the reactants and DI water)

**Procedure**
**Part A: Preparation of the Standard Solutions for the Calibration Curve**
Use the CV stock solution, 2.0 x $10^{-5}$ M, and pipet the appropriate volume into each test tubes. Pipet in the DI water, cover with Parafilm, and invert three times to mix.

Standard 1: Pipet 1.0 mL CV stock and 4.0 mL DI water

Standard 2: Pipet 2.0 mL CV stock and 3.0 mL DI water

Standard 3: Pipet 3.0 mL CV stock and 2.0 mL DI water

Standard 4: Pipet 4.0 mL CV stock and 1.0 mL DI water

Set the wavelength on the spectrophotometer to 590 nm, use deionized water as the blank, and measure and record the absorbance of each of the standard solutions. This data will be used to prepare a calibration curve.

**Part B: Determining the Exponents in the Rate Law Equation**
Use the crystal violet stock solution, 2.0 x $10^{-5}$ M, and the NaOH stock solution, 0.10 M

**Table 1: Volumes of Reactants for the Rate Experiment**

| Trial Number | Volume CV soln, mL | Volume DI $H_2O$, mL | Volume NaOH soln, mL |
|---|---|---|---|
| 1 | 1.0 | 1.0 | 3.0 |
| 2 | 2.0 | 0.0 | 3.0 |
| 3 | 2.0 | 2.0 | 1.0 |

***Do one trial at a time; start to finish. Put the reactants into the test tube before placing the test tube into the spectrophotometer sample compartment.***

a) Add deionized water to a test tube and use it as a blank to zero the spectrophotometer.
b) Pipet the crystal violet solution into a test tube, then pipet the deionized water into the test tube.
c) Wipe the outside of the test tube with a Kimwipe.
d) Pipet the NaOH into the test tube, and start the timer when you first start to pipet the NaOH into the test tube.
e) Place a piece of Parafilm over the test tube and invert three times to mix.

f) Remove the Parafilm, and immediately place the test tube into the sample compartment of the spectrophotometer, and then close the lid.
g) Record the absorbance every 10 seconds for 240 seconds. The first time point that you will probably be able to record data for is the 20 or 30 second time.
h) Perform steps a-g for all three trials, using the volumes indicated in Table 1.

**<u>Repeat Trials 1 – 3 for a second time</u>, which will let you average the absorbances.** There will be trial 1a, 1b, 2a, 2b, 3a, 3b, and then the calculated averages: $1_{ave}$, $2_{ave}$, $3_{ave}$ in your spreadsheet.

**Calculations:** *(Each calculation is relatively easy, but there are a lot of them. There is a calculation video in Bb to help you.)*

1) Calculate the concentration of $CV^{1+}$ in each standard solution. (Mc*Vc = Md*Vd)
2) Calculate the initial concentration of $CV^{1+}$ in each rate experiment trial. (Mc*Vc = Md*Vd) The total volume in each test tube was 5.0 mL.
3) Calculate the initial concentration of NaOH in each rate experiment trial. (Mc*Vc = Md*Vd) The total volume in each test tube was 5.0 mL.
4) Calculate the average absorbance for each time point for each trial. (Use Excel or Google Sheets for these calculations. There is a video in Bb.)
5) Use Excel or Google Sheets to make a calibration curve with the concentrations of the standard solutions and their absorbances. Get the equation of the best-fit line, which will be in the y = mx + b format. The $R^2$ value and b will show you how well you made your standard solutions.
6) Calculate the average concentration for each time point, for each trial, using the equation of the best-fit line from the calibration curve and the average absorbance values. Use Excel or Google Sheets to do these calculations (watch the video in Blackboard).
7) Calculate the initial reaction rate for each trial; use $\Delta[CV^{1+}] / \Delta t$ with the first two time points you were able to record absorbance data for. Use the same two time points for all of the trials.
8) Determine the exponents in the rate law equation, by determining the ratio of concentrations and ratio of initial reaction rates, using the appropriate two trials from the rate experiments.
9) Confirm the order of $CV^{1+}$ you calculated by graphing ( ln $[CV^{1+}]$ vs. time (s) ); t is the x-axis. A straight line confirms a first order for CV. Make a graph of ( $(1/[CV^{1+}])$ vs. time (s) ). A straight line confirms a second order for CV.
10) Calculate the value of the rate constant, k, using the rate law equation you have determined so far and each trial in the rate experiments. Calculate the average k value.
11) Report out the rate law equation that you determined; use the average k value and the exponents you calculated.

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## Experiment 24: Systems in Equilibrium

*(This experiment was adapted from Santa Monica College, Chemistry 12, Properties of Systems in Equilibrium – Le Chatelier's Principle)*

**Purpose:** The purpose of this experiment is to study systems at equilibrium and equilibriums that have been disrupted.

**Background:** The concentrations of reactants and products at equilibrium are constant as a function of time. Thus, for a homogeneous aqueous system of the form

$$a\text{A}\,(aq) + b\text{B}\,(aq) \rightarrow c\text{C}\,(aq) + d\text{D}\,(aq) \qquad (1)$$

we can express the equilibrium-constant expression for this reaction as,

$$K_c = \frac{[\text{C}]^c\,[\text{D}]^d}{[\text{A}]^a\,[\text{B}]^b} \qquad (2)$$

It has been observed that when a reaction at equilibrium is disturbed by applying a stress, the reaction will respond by shifting its equilibrium position so as to counteract the effect of the stress. In other words, the concentrations of the reactants and products will shift so that the relationship described by Equation (2) is again satisfied. This idea was first proposed by Henri-Louis Le Châtelier and has since been referred to as, "Le Châtelier's Principle".
Note that when a reaction makes more products as a response to the disturbance, we call it a right-shift. When a reaction makes more reactants in response to the disturbance, we call it a left-shift.

For chemical reactions at equilibrium in aqueous solution, the most common types of disturbances (stresses) include changing the concentration of one of the aqueous solutes, changing the concentrations of all aqueous solutes by changing the total solution volume, or changing the temperature. The general responses of an aqueous system to these particular disturbances (perturbations) are tabulated below.

| Perturbation | Effect on Equilibrium Position | Effect on $K_c$ |
|---|---|---|
| Increase in concentration of a single reactant, or, decrease in concentration of a single product. | Shift to the right | None |
| Decrease in concentration of a single reactant, or, increase in concentration of a single product. | Shift to the left | None |
| Decrease in all aqueous concentrations due to an increase in solution volume resulting from the addition of solvent | Shift towards side with more solute particles | None |
| Increase in all aqueous concentrations due to a decrease in solution volume resulting from the removal of solvent (evaporation) | Shift towards side with less solute particles | None |
| Increase temperature of an exothermic reaction | Shift to the left | Decrease |
| Decrease temperature of an exothermic reaction | Shift to the right | Increase |
| Increase temperature of an endothermic reaction | Shift to the right | Increase |
| Decrease temperature of an endothermic reaction | Shift to the left | Decrease |
| Addition of an inert substance, catalyst, pure liquid, or pure solid | None | None |

In this experiment you will disturb reactions that have attained equilibrium. You will then observe how each reaction responds to that disturbance in order to restore equilibrium. In your notebook, describe these changes in terms of Le Châtelier's Principle. Show the chemical equations when you are explaining the changes.

**Part A – Acid-Base Equilibrium**

Here you will use **coupled equilibria** to change the equilibrium position of an acid-base reaction. In order to understand how coupled equilibria work, consider the reactions described by the chemical equations below (both reactions are in the same beaker):

$$A\,(aq) \rightarrow B\,(aq) \qquad \text{eqn. (1)}$$

$$B\,(aq) + C\,(aq) \rightarrow D\,(aq) \qquad \text{eqn. (2)}$$

Notice that the species B (*aq*) is common to both reactions. The presence of this common species couples these two reactions.

We can disturb the equilibrium position of equation (2) by the addition of some C (*aq*). The addition of C (*aq*) will cause the equilibrium position of equation (2) to shift right. This right shift in the equilibrium position of equation (2) will also result a corresponding decrease in the concentration of B (*aq*). Because B (*aq*) is also present in equation (1), the decrease in the concentration of B (*aq*) will in turn result in a right shift in the equilibrium position of equation (1). Thus, the addition of C (*aq*) to equation (2) actually results in a right shift in the equilibrium position of equation (1) because the equilibria are coupled.

In Part A we will observe the effect of various solutes on an acid-base indicator (a weak acid) at equilibrium. The equilibrium system can be written in the general form

$$HA\,(aq) \rightarrow H+\,(aq) + A–\,(aq) \qquad \text{eqn. (3)}$$

The equilibrium-constant expression for this reaction is

$$K_a = \frac{[H^+][A^-]}{[HA]} \qquad \text{eqn. (4)}$$

In this experiment, HA and $A^-$, are the acidic and basic forms of the indicator bromothymol blue. Since the two forms are different colors, you will be able to determine which form is predominant in the equilibrium mixture. In other words, you will be able to determine whether the equilibrium position lies to the left or to the right. Your goal will be to find a reagent that will shift the position of this equilibrium to the opposite side, and then another reagent that will shift it back towards its original position. Instead of directly adding HA or $A^-$ to the system, you will affect these shifts by adding $H^+$ or $OH^-$. Note that in order to determine the effect of $OH^-$ we must consider a second chemical reaction that shares a common species with the Reaction (3). The second reaction is the autoionization of water, which can be described by the equation

$$H_2O\,(l) \rightarrow H^+\,(aq) + OH^-\,(aq) \qquad (5)$$

Because Reactions (3) and (5) share a common chemical species (H+), you can use the concept of coupled equilibria to shift the equilibrium position of Reaction (3) by increasing or decreasing the concentration of $OH^-$ (*aq*).

**Part B – Solubility Equilibrium**

Here you will test the effects of changing temperature on the solubility of a salt. This type of equilibrium is often called a **solubility equilibrium** because it is written in the direction of the dissolution of the solid, as shown in the following example:

$$KNO_3\,(s) \rightarrow K^{1+}\,(aq) + NO_3^{1-}\,(aq) \qquad \text{eqn. (6)}$$

You will use an ionic compound, $KNO_3$, that is soluble at one temperature, but insoluble at another temperature.

**Part C – Complex Ion Equilibrium**

Certain metal ions, most often transition metals, exist in solution as complex ions in combination with other ions or molecules, called ligands. Common ligands include $H_2O$, $NH_3$, $Cl^-$ and $OH^-$. Many of these complex ions exhibit vibrant colors in solution. You will use $[Cu(H_2O)_6]^{2+}_{(aq)} + 4NH_{3\,(aq)} \rightleftharpoons [Cu(NH_3)_4(H_2O)_2]^{2+}_{(aq)} + 4H_2O_{(l)}$ (eqn. 7) reaction in this part.

**Part D – Dissolving Insoluble Solids**

In Part D you will use coupled equilibria to affect the solubility equilibrium of $Zn(OH)_{2(s)}$. The solubility equilibrium can be described by the equation

$$Zn(OH)_{2\,(s)} \rightleftharpoons Zn^{2+}\,_{(aq)} + 2\,OH^-\,_{(aq)} \qquad \text{eqn. (8)}$$

Now consider the reactions described by the following chemical equations, each of which shares a common species with the reaction (8):

$$H_2O\,_{(l)} \rightleftharpoons H^+\,_{(aq)} + OH^-\,_{(aq)} \qquad K_w = 1 \times 10^{-14} \qquad \text{eqn. (9)}$$

$$Zn^{2+}\,_{(aq)} + 4\,OH^-\,_{(aq)} \rightleftharpoons Zn(OH)_4^{2-}\,_{(aq)} \qquad K_f = 3 \times 10^{15} \qquad \text{eqn. (10)}$$

$$Zn^{2+}\,_{(aq)} + 4\,NH_{3\,(aq)} \rightleftharpoons Zn(NH_3)_4^{2+}\,_{(aq)} \qquad K_f = 1 \times 10^{9} \qquad \text{eqn. (11)}$$

Because equations (9), (10), and (11) each share a common species with equation (8) they can be coupled together. In Part D of this experiment you will observe the effect of coupling each of these equilibria on the solubility of $Zn(OH)_{2\,(s)}$. *(Notice that the products in equations 9, 10, and 11 are all aqueous.)*

**Chemicals**

| | | |
|---|---|---|
| Bromothymol blue | 6 M HCl | 6 M NaOH |
| $KNO_3$, solid | 0.1 M $CuSO_4$ | 6 M $NH_3$ |
| 0.1 M $Mg(NO_3)_2$ | 0.1 M $Zn(NO_3)_2$ | |

**Equipment**

| | | |
|---|---|---|
| 6 large test tubes | Test tube rack | Stirring rod |
| Plastic droppers | 10-mL graduated cylinder | Ice |
| Beakers | Hot plate | |

**Safety and Waste Disposal**
6 M HCl, 6 M NaOH, and 6 M $NH_3$ are extremely caustic and great care must be taken to avoid contact with eyes or skin. The bottles should not be removed from the fume hood.

**Procedure**

**Part A: Acid-Base Equilibrium**
Here you will find a reagent that will shift the acid-base equilibrium given by equation (3) in one direction and then a second reagent that will cause the equilibrium position to shift back in the opposite direction.
Reagents needed for this part are: deionized water, bromothymol blue solution, a 6 M HCl and a 6 M NaOH.

1. Add approximately 5 mL of deionized water to each of two large test tubes. Add 4 drops of the bromothymol blue indicator solution to each test tube. Report the color of your solution on your data sheet. Keep one of these test tubes as the original reference point.

2. Your solution from Step 1 currently contains one form of bromothymol blue (see background). Now predict which of the two 6 M reagents, the strong acid or the strong base, will cause a color change in your solution by making the bromothymol blue indicator shift to its other form. Add the 6 M reagent of your choice drop-by-drop and if your solution changes color, write the color of the solution and formula of the reagent in your notebook. If the addition of your reagent does not result in a color change, try the other reagent until you are successful.

3. Since equilibrium systems are reversible, it is possible to shift a reaction left or right repeatedly by changing the conditions. Now use the other 6 M reagent that will cause your solution from Step 2 to revert back to its original color (but diluted in intensity). Add the 6 M reagent drop-by-drop, mix well, and record your observation in your notebook. You should be able to get the color the change from one to the other (yellow to blue or blue to yellow), and then back again.

**Part B: Solubility Equilibrium**
You will test the effects of temperature change on the solubility of potassium nitrate. The solubility of $KNO_3$ increases as the temperature of the water is increased.
Reagents needed: solid $KNO_3$ and deionized water

1. Use the hot-water bath (50-60°C) set up in the hood for steps 3 and 5. Set up an ice bath using a 250 mL beaker, tap water and ice at your bench. You will need this for step 4.

2. Put 5.0 mL of room temperature deionized water into a large test tube. Measure 1.600 g of $KNO_3$ on tared weighing paper at the balance, and add this to the DI water in the large test tube. Cover with Parafilm and mix well. Record your observations.

3. Place this test tube in the hot-water bath (50-60°C). Make your observations as the contents of the test tube get warmed. Mix well and be patient.

4. Transfer this test tube to the ice-water bath. Make your observations as the contents of the test tube reach the temperature of the ice water. Mix well and be patient.

5. Transfer this test tube back to the hot-water bath. Make your observations as the contents of the test tube reach the temperature of the hot water. Mix well and be patient.

6. Dispose of the potassium nitrate solution in the waste bottle. Rinse and reuse the large test tube as needed.

**Part C: Complex Ion Equilibrium** *(use the Part C waste bottle)*
Here you will observe the formation of a complex ion equilibrium between $Cu^{2+}$ and $NH_3$ as in equation (7).
Reagents needed for this part are: 0.1 M $Cu^{2+}$, 6 M $NH_3$, and deionized water.

1. Put 5 mL of 0.1 M $Cu^{2+}$ solution into a large test tube. Record the color of this original solution. *(Note: $Cu^{2+}$ is actually $[Cu(H_2O)_6]^{2+}_{(aq)}$.)* Working in the fume hood, carefully add 3 drops of 6 M $NH_{3(aq)}$ Record the color of this solution. Add enough $NH_3$ to make the original solution change from clear light blue to dark blue with a precipitate. Add a few more drops of $NH_3$ to make the precipitate dissolve. Record all of your observations and number of drops used.

$Cu(OH)_{2(s)} \rightleftharpoons Cu^{2+}_{(aq)} + 2\ OH^{1-}_{(aq)}$ $NH_{3(aq)} + H_2O_{(l)} \rightleftharpoons NH_4^{1+}{}_{(aq)} + OH^{1-}_{(aq)}$

**Part D: Dissolving Insoluble Solids**
Here you will further examine how one reaction can affect the behavior of another reaction when the reactions share one or more common chemical species.
Reagents needed are: 0.1 M $Zn(NO_3)_{2\ (aq)}$, 0.1 M $Mg(NO_3)_{2\ (aq)}$, 6 M NaOH (*aq*), 6 M HCl (*aq*), and 6 M $NH_{3\ (aq)}$.

1. Label three large test tubes A, B, and C. Add about 2 mL of 0.1 M $Zn(NO_3)_2$ solution to each test tube. Add one drop of 6 M NaOH solution to each test tube. Mix each solution and record your observations. *(This will form a precipitate in each test tube.)*

2. Into test tube A, add 20 drops of 6 M HCl (*aq*) drop-by-drop while mixing. Record all observations.

3. Into test tube B, add 20 drops of 6 M NaOH (*aq*) drop-by-drop while mixing. Record all observations.

4. Into test tube C, add 20 drops of 6 M $NH_3$ (*aq*) drop-by-drop while mixing. Record all observations.

5. Label three additional large test tubes D, E, and F. Add about 2 mL of 0.1 M $Mg(NO_3)_2$ solution to each test tube. Add one drop of 6 M NaOH solution to each test tube. Mix each solution and record your observations.
*(This will form a precipitate in each test tube.)*

6. Into test tube D, add 20 drops of 6 M $HCl_{(aq)}$ drop-by-drop while mixing. Record all observations.

7. Into test tube E, add 20 drops of 6 M $NaOH_{(aq)}$ drop-by-drop while mixing. Record all observations.

8. Into test tube F, add 20 drops of 6 M $NH_{3(aq)}$ drop-by-drop while mixing. Record all observations.

**For the Data Tables section of your notebook:** Record all of your observations made in each part of this experiment. Neatly organize your observations, and provide a brief statement with each observation so someone reading your notebook can tell what was done experimentally that caused that observation.

**For the Results section of your notebook:** Explain each observation by referring to Le Châtelier's Principle, the disturbances, shifts to the right, shifts to the left, and why. *Include the chemical equations*.

**For the Conclusion section of your notebook:** Comment on whether the results matched up with what you expected based on Le Châtelier's Principle. Try to explain any discrepancies.

## Experiment 25: Determination of $K_c$ for a Chemical Reaction

*This experiment was adapted from **Ross S. Nord**, Chemistry Department, Eastern Michigan University.*

**Purpose:** To determine the equilibrium constant for the reaction between iron(III) and thiocyanate, and to study the equilibrium system of this chemical reaction.

**Background:** Iron(III) and thiocyanate react to form an equilibrium mixture with the product formed; the iron-thiocyanate complex ion. The net-ionic equation is:

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

The iron(III) solution is slightly pale-yellow in color, the thiocyanate solution in colorless, and the complex ion product is an orange-red color. The colored product will allow for the use of a spectrophotometer to quantitate the amount of product made. Using the data from the spectrophotometer, along with concentrations of iron(III) and thiocyanate, we will study this equilibrium system.

When a reaction reaches equilibrium, not all of the reactants became product. A mixture of reactants and products exist. The equilibrium constant is calculated as the ratio of the concentration of product(s) divided by the concentration of reactant(s). This is the simplified version of the math. The more complicated version involves calculations with activity coefficients and ionic strength due to the fact that this equilibrium system involves ionic species. Overall, we can say that it is typical for the math to be simplified for the calculation of the equilibrium constant.

$$Kc = \frac{[FeNCS^{2+}]}{([Fe^{3+}] \times [SCN^{1-}])}$$

This experiment consists of three parts:

**Part 1:** Determine the $\lambda_{max}$ for the $FeNCS^{2+}$ ion. Since the solution is an orange-reddish color, wavelengths in the 400 nm to 500 nm range are being absorbed.

**Part 2:** The reaction between the iron(III) and thiocyanate will be studied as an equilibrium, with the ion concentration as low as possible. Reasonable amounts of each reactant will be combined to allow the equilibrium mixture to form.

**Part 3:** The reaction between the iron(III) and thiocyanate will be studied as an equilibrium, with the ion concentration increased by the addition of 0.10 M $KNO_3$. The amount of each reactant will be the same as in Part 2.

**Chemicals** *(Use caution with each of these solutions.)*
0.0020 M $Fe(NO_3)_3$ in 0.0050 M $HNO_3$
0.0020 M NaSCN in DI water
0.10 M $KNO_3$
Deionized water

**Equipment**
1.0 mL pipet (1), 10.0 mL pipet (2), pipettor (2), beakers (3)
Test tubes, Test tube rack, Spectrophotometer
Parafilm

**Procedure**
**Part 1: Determining $\lambda_{max}$ for $FeNCS^{2+}$**

a) Combine 1.50 mL of $Fe^{3+}$ with 2.00 mL of $SCN^{1-}$ in a small test tube.
b) Cover the test tube with Parafilm. Secure your thumb over the Parafilm and invert the test tube to mix three times. This is the equilibrium mixture.
c) Set the software on the spectrophotometer to perform a wavelength scan.
d) Place a test tube of $Fe^{3+}$ solution in the sample compartment of the spectrophotometer as the blank and zero the spectrophotometer.
e) Place the test tube with the equilibrium mixture in the sample compartment and run the wavelength scan from 400 nm to 700 nm.
f) The spectrophotometer will display a graph of wavelength vs. absorbance. Record the wavelength of maximum absorbance.

**Part 2: Observe the Equilibrium Mixture with Low Ionic Strength**

a) Prepare a series of equilibrium mixtures, each in its own small test tube. Use the volumes of each reactant listed in Table 1.
b) Cover each test tube with Parafilm. Secure the film with your thumb and invert to mix three times.
c) Set the spectrophotometer wavelength to $\lambda_{max}$. Use the $Fe^{3+}$ solution as your blank and zero the spectrophotometer.
d) Measure the absorbance of each equilibrium mixture.
e) Add 1.5 mL of DI water to each test tube and blank, and invert to mix three times.
f) Rezero with the blank and measure the absorbance of each equilibrium mixture.
g) Add 1.0 mL of DI water to each test tube and blank, and invert to mix three times.
h) Rezero with the blank and measure the absorbance of each equilibrium mixture.

**Table 1 Solution Preparation for Part 2**

| Solution Number | Volume of $Fe^{3+}$, mL | Volume of $SCN^{1-}$, mL |
|---|---|---|
| Blank | 4 | 0 |
| 1 | 0.50 | 3.00 |
| 2 | 1.00 | 2.50 |
| 3 | 1.50 | 2.00 |
| 4 | 2.00 | 1.50 |
| 5 | 2.50 | 1.00 |
| 6 | 3.00 | 0.50 |

## Part 3: Observe the Equilibrium Mixture with High Ionic Strength

a) Prepare a series of equilibrium mixtures, each in its own small test tube. Use the volumes of each reactant listed in Table 1 (same as in Part 1).
b) Cover each test tube with Parafilm. Secure the film with your thumb and invert to mix three times.
c) Set the spectrophotometer wavelength to $\lambda_{max}$. Use the $Fe^{3+}$ solution as your blank and zero the spectrophotometer.
d) Measure the absorbance of each equilibrium mixture.
e) Add 1.5 mL of 0.10 M $KNO_3$ to each test tube and blank, and invert to mix three times.
f) Rezero with the blank and measure the absorbance of each equilibrium mixture.
g) Add 1.0 mL of 0.10 M $KNO_3$ to each test tube and blank, and invert to mix three times.
h) Rezero with the blank and measure the absorbance of each equilibrium mixture.

## Calculations

Use Excel or Google Sheets for the calculations. This will save a lot of time and provide experience with programming spreadsheets. There is a video in Blackboard that shows you how to make the Excel / Google Sheets file for this experiment.

**You will have to calculate the following:**

a) The initial concentration of iron(III).
b) The initial concentration of thiocyanate.
c) The concentration of $FeNCS^{2+}$ made (this is the equilibrium concentration). *(Consult the prelab PowerPoint file.)*
d) The equilibrium concentration of iron(III).
e) The equilibrium concentration of thiocyanate.
f) The value of the equilibrium constant, using equilibrium concentrations.
g) The average Kc.

*All of these calculations are shown in the prelab PowerPoint file and in the video.*

## Conclusion

When you write your conclusion, consider these topics:

a) Was the equilibrium constant a constant within each table, considering experimental error?
b) Was the equilibrium constant a constant after the dilution of water, considering experimental error?
c) Was the equilibrium constant affected by the addition of $KNO_3$, more so than the addition of DI water?
d) The preparation of the original solutions was the same in Part 2 and Part 3. How do those equilibrium constant values compare?
e) Did you notice any patterns with the data?

## Experiment 26: Titration of a Commercial Antacid

*(This experiment was adapted from CUNY.)*

### Purpose

To determine the amount of $CaCO_3$ present in a commercial antacid tablet.

### Background

The parietal cells in the stomach secrete hydrochloric acid (HCl) at a concentration of roughly 0.16 M. The flow of HCl increases when food enters the stomach. If you eat or drink too much, you may develop heartburn or indigestion. Antacids are used to neutralize this excess acid.

The active ingredient in antacids is calcium carbonate, $CaCO_3$, a base. There are also other ingredients, such as binders and flavors present in each tablet.

HCl is neutralized by calcium carbonate as illustrated below:

$$CaCO_{3\,(aq)} + H^{+}_{(aq)} \rightleftharpoons Ca^{2+}(aq) + HCO_3^{1-}{}_{(aq)}$$

$$HCO_3^{1-}{}_{(aq)} + H^{+}_{(aq)} \rightleftharpoons H_2CO_{3(aq)} \rightleftharpoons CO_2\,(g) + H_2O\,(l)$$

To determine the quantity of $CaCO_3$ in the antacid tablet, we are first going to dissolve the tablet in an excess amount of acid, HCl, of known concentration. Some of the HCl will be neutralized by the carbonate, but there will be some remaining. We will then perform a titration with NaOH to figure out the amount of excess HCl left over. Then, from this, we can calculate how much acid reacted with the antacid, followed by the amount of antacid present in the mass of tablet used. This method of analysis is called a back-titration. (Note: start – left over = used by $CaCO_3$)

The reactions above are reversible, which means that the $CO_2$ dissolved in water would produce some carbonic acid. This acid would react with the NaOH we are titrating and give us inaccurate results. Therefore, it is important to boil the solution when the calcium carbonate reacts with acid, to remove $CO_2$ as a gas. The net result is below:

$$CaCO_{3(aq)} + 2\,HCl_{(aq)} \rightarrow CaCl_{2(aq)} + CO_{2(g)}(\uparrow) + H_2O_{(l)}$$

### Chemicals

0.10 M hydrochloric acid, HCl
0.10 M sodium hydroxide, NaOH
Antacid tablets (label states 500 mg $CaCO_3$ per tablet)
Phenolphthalein, 1%
Deionized water

### Equipment

| | |
|---|---|
| Buret, buret stand and clamp | Mortar & Pestle |
| Erlenmeyer flask, 125 mL | Beakers, as needed |
| Graduated cylinder, 50 mL or 100 mL | Stirring rod |
| Hot plate (use the one in the hood) | Balance and weigh paper |

**Procedure** *(Do one trial at a time, start to finish.)*

1. Rinse your buret with DI $H_2O$, and then rinse it three times with 2 mL-portions of the NaOH solution. Fill the buret with the 0.10 M NaOH solution. Allow enough of the NaOH solution to drain from the buret to remove the air bubble in the tip of the buret. Refill the buret as needed.

2. There will be hot-water baths set up in the fume hoods. Let the bath water get hot enough to boil gently. You will be using this to heat your acid and antacid mixture.

3. Obtain two antacid tablets. Find their exact, combined mass with the lab balance.

4. Crush both tablets with a mortar and pestle, until they are a fine powder.

5. Use the graduated cylinder to add 50.0 mL of the 0.10 M HCl to the 125 mL Erlenmeyer flask. Rinse the graduated cylinder with 10 mL of DI water and add this rinse to the Erlenmeyer flask.

6. Determine the exact mass of approximately 0.450 g of this powder. Your sample mass can be slightly less than 0.450 g, but do not use more than 0.450 g. Transfer this sample into the Erlenmeyer flask.

7. Swirl the flask to dissolve the antacid power in the acid and place this flask in the hot-water bath for 10 minutes. After the 10 minutes, remove the Erlenmeyer flask from the hot-water bath and allow it to cool on your lab bench. Placing the Erlenmeyer in a cool-water bath will help cool it faster.

8. Add three drops of phenolphthalein indicator to the Erlenmeyer flask. Mix well.

9. Record the initial buret volume. Titrate the solution in the flask until the endpoint is reached. The endpoint will be visible when the indicator turns very faint pink. Record the final buret volume.

10. Repeat steps 5 – 9 for two more trials. You should have a total of 3 useable trials. Do one trial at a time, from start to finish.

**Data Needed**

Exact mass of two antacid tablets

For each trial, make sure you record the following information:

a) Volume of HCl added into the Erlenmeyer flask

b) Exact mass of antacid powder added to the Erlenmeyer flask

c) The initial volume of NaOH (before the titration is started)

d) The final volume of NaOH (after the titration is stopped)

**Calculations**

For each trial, calculate the following: (remember, M=moles/L so M x L = moles)

a) Calculate the number of moles of HCl added into the Erlenmeyer flask
b) Calculate the number of moles of NaOH delivered from the buret
c) Subtract the moles NaOH from the moles HCl; this difference is the moles of HCl used to neutralize the $CaCO_3$
d) Calculate the moles $CaCO_3$. remember (2 $H^+$:1 $CO_3^{2-}$)
e) Convert the moles of $CaCO_3$ in the flask to grams of $CaCO_3$
f) Divide the grams of $CaCO_3$ by the mass of the antacid powder put into the flask; (g $CaCO_3$ / g antacid *powder in flask*)
g) Divide the mass of the two antacid tablets by two; this gives the g antacid powder per one tablet
h) Calculate the grams $CaCO_3$ / tablet with the following calculation:

$$\frac{\text{g CaCO}_3}{\text{g antacid powder in flask}} \times \frac{\text{g antacid powder}}{\text{one tablet}} = \frac{\text{g CaCO}_3}{\text{tablet}}$$

***(f) x (g) = (h)***

i) Average the three values of the g $CaCO_3$ / tablet
j) How does this average g/tablet compare to the manufacture's claim? Calculate the percent difference between your experimental average result and the label value of antacid. Use the average experimental result for this calculation.
A % difference of less than 5% is good.

$$\%\ \text{difference} = \frac{/\ \text{true g/tablet} - \text{exp g/tablet}\ /}{\text{true g/tablet}} \times 100$$

*the true g/tablet is the value from the bottle

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## Experiment 27: Qualitative Analysis of Anions

*(This experiment was adapted from Santa Monica College, CHEM 12.)*

### Purpose

The purpose of this experiment is to use chemical tests to identify common anions in an aqueous solution. This will reinforce the technique of writing and balancing chemical reactions, many of which are acid-base reactions.

### Background

In this experiment you will use qualitative analysis to identify the various anions in a sample. Specifically, you will test for the presence of each of the following anions:

$CO_3^{2-}$, $SO_4^{2-}$, $PO_4^{3-}$, $SCN^{1-}$, $Cl^{1-}$, $NO_3^{1-}$

The methodology used in identifying the anions will involve the use of a small portion of the unknown mixture (1 mL) to perform a test for each anion individually.

In some cases, the test for a particular ion will be complicated by the presence of other ions in the mixture that will interfere with the test. In these situations, the interfering ions must be removed before the test can be performed.

### Chemicals

| | | | |
|---|---|---|---|
| 1 M $Na_2CO_3$ | 0.5 M $Na_2SO_4$ | 1 M $BaCl_2$ | 0.5 M $Na_2HPO_4$ |
| 0.5 M $(NH_4)_2MoO_4$ | 0.5 M NaSCN | 0.1 M $Fe(NO_3)_3$ | 0.5 M NaCl |
| 0.1 M $AgNO_3$ | 0.5 M $NaNO_3$ | 1 M $CuSO_4$ | 6 M HCl |
| 6 M $HNO_3$ | 1 M acetic acid | 6 M NaOH | Aluminum granules |
| Unknown 1 | Unknown 2 | | |

### Equipment

| | | | |
|---|---|---|---|
| 4 test tubes (rinse & reuse) | glass stirring rod | 10-mL grad. cylinder | red litmus paper |
| Plastic droppers | Hot water bath (hood) | blue litmus paper | Centrifuge |
| Parafilm | | | |

### General Instructions

For each anion you will perform:

1) a positive control test on 1 mL of solution of the anion
2) a positive control test on 1 mL of a diluted solution of the anion. (The diluted anion solutions can be prepared by adding 10 drops of deionized water to 10 drops of the anion solution. This will yield approximately 1 mL of a diluted solution.)
3) the same test on 1 mL of each unknown solution, which will contain at least one of the anions in the control solutions.

In the Raw Data section of your notebook, record the anion test and observations in your notebook. Tabulate your observations using the following format:

| **Test Tube #** | 1 | 2 | 3 | 4 |
|---|---|---|---|---|
| **Anion being Tested** | Control Test Observation | Diluted Control Test Observation | Unknown Solution 1 | Unknown Solution 2 |

Perform these specific anion tests:
The specific anion tests are:

**Test for the presence of carbonate ion, $CO_3^{2-}$**

| **Test Tube** | Control | Diluted Control | Unknown 1 | Unknown 2 |
|---|---|---|---|---|
| **Anion being Tested $CO_3^{2-}$** | Add 1 mL of 1 M $Na_2CO_3$ | Add 10 drops of 1 M $Na_2CO_3$ and 10 drops of DI $H_2O$ | Add 1 mL of Unknown 1 | Add 1 mL of Unknown 2 |

Add 1 mL of 6 M HCl to each test tube. Effervescence indicates the presence of $CO_3^{2-}$. In the concentrated control solution, you should see effervescence for at least a few seconds.
It may be necessary to place the diluted test tube in the hot water bath (in the hood) in order to observe the effervescence.
For the unknowns, if no effervescence is observed, place the test tubes in the hot water bath before concluding whether $CO_3^{2-}$ is not present.

**Test for the presence of sulfate ion, $SO_4^{2-}$**

| **Test Tube** | Control | Diluted Control | Unknown 1 | Unknown 2 |
|---|---|---|---|---|
| **Anion being Tested $SO_4^{2-}$** | Add 1 mL of 0.5 M $Na_2SO_4$ | Add 10 drops of 0.5 M $Na_2SO_4$ and 10 drops of DI $H_2O$ | Add 1 mL of Unknown 1 | Add 1 mL of Unknown 2 |

Add 1 mL of 6 M HCl to each unknown's test tube if the unknown contains the carbonate ion. This will remove the carbonate anion as $CO_2$ gas.
Next add 3 drops of 1 M $BaCl_2$ to each test tube. A finely divided, white precipitate indicates the presence of the $SO_4^{2-}$ ion.
Use your observations to conclude whether $SO_4^{2-}$ is present or not in each unknown.

**Test for the presence of phosphate ion, $PO_4^{3-}$**

| **Test Tube** | Control | Diluted Control | Unknown 1 | Unknown 2 |
|---|---|---|---|---|
| **Anion being Tested $PO_4^{3-}$** | Add 1 mL of 0.5 M $Na_2HPO_4$ | Add 10 drops of 0.5 M $Na_2HPO_4$ and 10 drops of DI $H_2O$ | Add 1 mL of Unknown 1 | Add 1 mL of Unknown 2 |

Add 1 ml of the ammonium molybdate reagent to each test tube. Add 2 drops of the tin chloride reagent (tin chloride in glycerol) and observe the color. (You may have to wait a few minutes for the color to form.) An intense blue color indicates the presence of the phosphate ion. Use your observations to conclude whether $PO_4^{3-}$ is present or not in each unknown.

**Test for the presence of thiocyanate ion, $SCN^{1-}$**

| **Test Tube** | Control | Diluted Control | Unknown 1 | Unknown 2 |
|---|---|---|---|---|
| **Anion being Tested $SCN^{1-}$** | Add 1 mL of 0.5 M NaSCN | Add 10 drops of 0.5 M NaSCN and 10 drops of DI $H_2O$ | Add 1 mL of Unknown 1 | Add 1 mL of Unknown 2 |

Add 1 mL of acetic acid to each unknown's test tube if the unknown contains the carbonate ion. This will remove the carbonate anion as $CO_2$ gas. **Then see the note below.

Now add 2 drops of 0.1 M $Fe(NO_3)_3$. A dark red solution indicates the presence of $SCN^{1-}$ (as $FeNCS^{2+}_{(aq)}$)

Use your observations to conclude whether $SCN^{1-}$ is present or not in each unknown.

****Important:** If your unknown contains $PO_4^{3-}$ it will interfere with the test for the $SCN^{1-}$ since $PO_4^{3-}$ will form a precipitate with $Fe^{3+}$ (white, greyish, or pink solid).

$$Fe^{3+}_{(aq)} + PO_4^{3-}_{(aq)} \rightarrow FePO_{4(s)}$$

In this case it will be necessary to remove all of the $PO_4^{3-}$ before any conclusion can be made concerning the presence of $SCN^{1-}$. The $PO_4^{3-}$ (as $FePO_{4(s)}$) can be removed by centrifuging the mixture and decanting the supernatant solution. Now add $Fe(NO_3)_3$ to the supernatant solution. If more precipitate forms, centrifuge and decant a second time. Now test for the presence of thiocyanate ion in the supernatant solution with the procedure above.

**Test for the presence of chloride ion, $Cl^{1-}$**

| **Test Tube** | Control | Diluted Control | Unknown 1 | Unknown 2 |
|---|---|---|---|---|
| **Anion being Tested $Cl^{1-}$** | Add 1 mL of 0.5 M NaCl | Add 10 drops of 0.5 M NaCl and 10 drops of DI $H_2O$ | Add 1 mL of Unknown 1 | Add 1 mL of Unknown 2 |

Add 1 mL of $HNO_3$ to each unknown's test tube if the unknown contains the carbonate ion. This will remove the carbonate anion as $CO_2$ gas. **Then see the note below.

Next add 2 to 3 drops of 0.1 M AgNO3. The formation of a white, curdy precipitate indicates the presence of $Cl^{1-}$. *(Curdy means small lumps.)*

Use your observations to conclude whether $Cl^{1-}$ is present or not in each unknown.

****Important:** If your unknown contains $SCN^{1-}$ it will interfere with the test for the $Cl^{1-}$ since it will form a white precipitate with $Ag^{1+}$.

$$Ag^{1+}_{(aq)} + SCN^{1-}_{(aq)} \rightarrow AgSCN_{(s)}$$

In this case put 1 mL of your unknown sample into a small 50 mL beaker and add 1 mL of 6 M $HNO_3$. Boil the solution very gently on the hotplate (in the hood) until the volume has decreased by about one half. Under these conditions the $SCN^{1-}$ will decompose. Now pour this solution into a test tube, and test for the presence of the $Cl^{1-}$ ion following the procedure above.

**Test for the presence of nitrate ion, $NO_3^{1-}$**

| **Test Tube** | Control | Diluted Control | Unknown 1 | Unknown 2 |
|---|---|---|---|---|
| **Anion being Tested $NO_3^{1-}$** | Add 1 mL of 0.5 M $NaNO_3$ | Add 10 drops of 0.5 M $NaNO_3$ and 10 drops of DI $H_2O$ | Add 1 mL of Unknown 1 | Add 1 mL of Unknown 2 |

Add 1 mL of 6 M NaOH to each test tube. Then add a few granules or one larger piece of aluminum metal and put the test tube in the hot water bath (in the hood).

The reaction between Al and NaOH will produce $H_2$ gas which will reduce the $NO_3^{1-}$ to $NH_3$. The $NH_3$ can be detected by placing a piece of moistened red litmus paper directly above (but not in contact with) the mouth of the test tube. If the red litmus paper turns blue (due to $NH_3$ vapor coming out of the test tube) then it can be concluded that $NO_3^{1-}$ is present in the unknown. Note that small blue spots produced on the red litmus paper are the result of spray from the basic solution in the test tube and do not necessarily indicate the presence of nitrate.

Use your observations to conclude whether $NO_3^{1-}$ is present or not in each unknown.

*This test is referred to as Devarda's test (Chemistry.Stackexchange.com):*

$$3\ NO_{3\,(aq)}^{1-} + 8\ Al_{(s)} + 5\ OH_{(aq)}^{1-} + 18\ H_2O_{(l)} \rightarrow 3\ NH_{3(g)} + 8\ [Al(OH)_4]_{(aq)}^{1-}$$

**Important:** If your unknown contains $SCN^{1-}$ it will interfere with the test for the $NO_3^{1-}$. $SCN^{1-}$ will give a false-positive result for $NO_3^{1-}$. In this case add 1 mL of your unknown to 1 mL of 1 M $CuSO_4$ in a test tube. Place the test tube in a hot water bath for about 2 minutes (in the hood). Centrifuge the mixture and decant the supernatant solution into another test tube. The solid may be discarded in the waste.
To the supernatant solution add 1 mL of 1 M $Na_2CO_3$. Centrifuge the mixture and decant 1 mL of the supernatant solution into another test tube. The solid may again be discarded in the waste.
Now test for the presence of nitrate ion by using the supernatant solution in the test tube and following the procedure above.

**In the Data Tables section of your notebook:**

1) Tabulate your results for the control test, diluted control test, unknown 1 and unknown 2. Use the following format.

| Anion being Tested | Control Test Observation | Diluted Control Test Observation | Unknown Solution 1 Obs. | Unknown Solution 2 Obs. |
|---|---|---|---|---|

**In the Calculations / Results section of your notebook:**

1) Use the chart on the next page for your two unknowns that includes all of the anion tests performed and your observations and conclusion about each unknown for each test. Cut-out and tape this chart into your notebook.
2) Write the balanced chemical equation for the carbonate test, sulfate test, thiocyanate test, and chloride ion test. (4 points total; 1 point per equation)

**In the Conclusion section of your notebook:**

1) Summarize the anions present in each of your two unknowns and the logic used to make your decisions.

Please use this chart in the Calculations / Results section of your notebook. Cut-out and tape this chart into your notebook.

| Anion Test Performed | Unknown 1 | Unknown 2 |
|---|---|---|
| **Test for $CO_3^{2-}$** Observation<br>----------<br>Conclusion (1 point) | ---------- | ---------- |
| **Test for $SO_4^{2-}$** Observation<br>----------<br>Conclusion (1 point) | ---------- | ---------- |
| **Test for $PO_4^{2-}$** Observation<br>----------<br>Conclusion (1 point) | ---------- | ---------- |
| **Test for $SCN^-$** Observation<br>----------<br>Conclusion (1 point) | ---------- | ---------- |
| **Test for $Cl^-$** Observation<br>----------<br>Conclusion (1 point) | ---------- | ---------- |
| **Test for $NO_3^-$** Observation<br>----------<br>Conclusion (1 point) | ---------- | ---------- |
| **Overall Conclusion:** List anions present in each unknown (2 points) | | |

Do not include information about interfering ion work.
Each conclusion for each unknown in the table above is worth 1 point.
Each overall conclusion is worth 2 points.
The table above is worth a possible 16 points.

Remember to write the four balanced equations in your notebook.

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## Experiment 28: Qualitative Analysis of Cations

*(This experiment was adapted from Colby College, Experiment 4.)*

### Purpose

The purpose of this experiment is to develop and use a chemical-test analysis scheme for the identification of cations.

### Background *(This is a fictional story.)*

Chemical Solutions Incorporated (CSI) has earned a contract from the City of Trenton, which is trying to identify the source of metal contamination detected in the Delaware River. Excessively high levels of the heavy metal cations $Ag^{+}$, $Cu^{2+}$, $Fe^{3+}$, $Zn^{2+}$, and $Ba^{2+}$ have been detected in the region of the Trenton State House. The businesses that are likely suspects for contributing one or more heavy metal cations to the river water are shown in Figure 1.

CSI's research team has compiled a useful summary about inorganic qualitative analysis, which can be used to detect ions present in a water sample. You will use these methods to separate and identify the presence (or absence) of the relevant cations in river water samples. Additionally, the City would like an easy to follow, effective qualitative analysis scheme that they can use to monitor the river water for these cations in the future.

Therefore, as an investigator, you will develop/use a qualitative analysis scheme to separate each cation and confirm its identity. This scheme will be used to test water samples taken from two of the nine possible pollution sites.

To familiarize yourself with these different chemical tests, you will first use known solutions to observe the characteristic behavior of each cation before testing the river samples. The two river samples are labeled as unknowns.

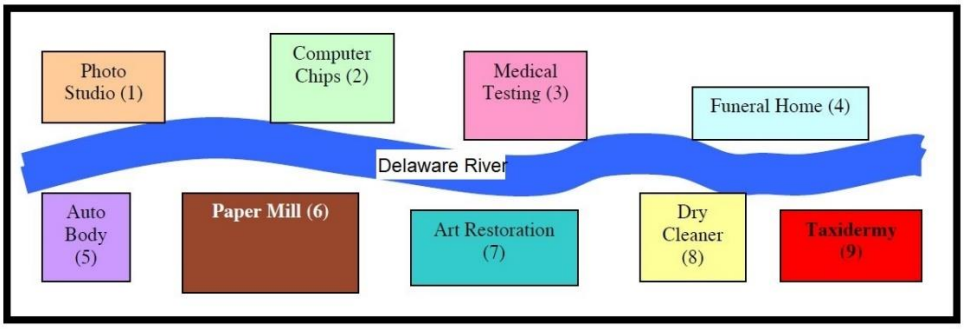



Figure 1. Map of the contaminated area of the Delaware River with possible rogue polluters.

## Qualitative Analysis Summary

### I. Sparingly Soluble Salts

Some cations form relatively insoluble salts that precipitate out of aqueous solution. For example, chloride salts are generally soluble with the notable exceptions of the salts containing $Ag^{+}$, $Pb^{+2}$, and $Hg_2^{2+}$. Similarly, most sulfate salts are soluble except $BaSO_4$, $PbSO_4$, $Hg_2SO_4$, $AgSO_4$, and $CaSO_4$.

**Therefore, the addition of $Cl^{1-}$ can be used to test for silver(I) cations and the addition of $SO_4^{2-}$ can be used to test for the silver(I) and barium cations.**

Most hydroxide salts are insoluble. For example, the solubility of $Fe^{3+}$ in basic solution is governed by the reaction shown in Eqn. 1. The equilibrium constant for the reaction $K_{sp}$ is $6.3 \times 10^{-38}$, which says that $Fe(OH)_3$ is sparingly soluble under most conditions. Indeed, for hydroxide concentrations greater than $10^{-11}$ M (pH > 3), $Fe^{3+}$ is relatively insoluble. Therefore, in a qualitative analysis scheme, $Fe^{3+}$ may be removed from solution by making the solution basic, thereby precipitating $Fe(OH)_3$. This precipitate can be removed from solution by centrifugation.

$$Fe(OH)_{3\,(s)} \rightleftharpoons Fe^{3+}_{(aq)} + 3\ OH^{1-}_{(aq)} \qquad \text{Eqn. (1)}$$

However, other cations for which you are testing also form insoluble hydroxides. Therefore, the addition of base to a mixture of cations may precipitate other metal hydroxides in addition to $Fe(OH)_3$. For example, if $Zn^{2+}$ is also present, the analogous reaction and relationships hold (eqn. 2). Copper also forms a hydroxide precipitate.

$$Zn(OH)_{2\,(s)} \rightleftharpoons Zn^{2+}_{(aq)} + 2\ OH^{1-}_{\ (aq)} \qquad \text{Eqn. (2)}$$

At hydroxide concentrations greater than $10^{-6}$ M (pH > 8), $Cu^{2+}$, $Fe^{3+}$ and $Zn^{2+}$ are insoluble and would coprecipitate as their hydroxides. In theory, one might be able to adjust the pH to the range where $Fe^{3+}$ will precipitate but $Zn^{2+}$ will not, but in practice this approach works poorly. Alternative approaches to separating $Fe^{3+}$ and $Zn^{2+}$ are either to use the amphoteric nature of $Zn(OH)_2$ ; the ability of $Zn^{2+}$ to form complex ions as described below.

**Therefore, the addition of $OH^{1-}$ can be used to form precipitates of copper(II), iron(III), silver(I), and zinc(II).**

### II. Amphoteric Hydroxides

Some metal-hydroxide precipitates dissolve in excess hydroxide solutions because of the formation of soluble hydroxide complex ions, as seen for $Zn^{2+}$ in eqn. 3. Such hydroxides are called amphoteric hydroxides because they will react with and dissolve in both acid and base. The equilibrium constant for this reaction is very product-favored ($K = 2 \times 10^{20}$). Therefore, $Zn(OH)_2$ solid will form when small amounts of a strong base are added to a $Zn^{2+}$ containing solution, <u>but the continued addition of strong base will dissolve that precipitate</u> as $[Zn(OH)_4]^{2-}_{(aq)}$ is formed. This amphoteric behavior is useful in qualitative analysis.

$Zn(OH)_{2\,(s)} + 2\ OH^{1-}{}_{(aq)} \rightleftharpoons [Zn(OH)_4]^{2-}{}_{(aq)}$ Eqn. (3)

**Therefore, the addition of excess $OH^{1-}$ can be used to separate the zinc(II) cation from the copper(II), iron(III), and silver(I) cations.**

## III. Complex Ion Formation

The formation of other complexes ions can also be useful. For example, $Cu^{2+}$ and ammonia react to form the complex ion $[Cu(NH_3)_4]^{2+}{}_{(aq)}$ (eqn. 4) with a large equilibrium constant ($K= 4.8 \times 10^{12}$), showing that the formation of $[Cu(NH_3)_4]^{2+}$ is very favorable. The utility of ammonia in qualitative analysis schemes can be shown by the behavior of a mixture of $Fe^{3+}$ and $Cu^{2+}$ ions. If concentrated ammonia (a basic solution because ammonia is a weak base (eqn. 5)) is added to a solution containing $Fe^{3+}$ and $Cu^{2+}$ ions, iron will precipitate in the presence of base (eqn. 1) and the soluble complex ion $[Cu(NH_3)_4]^{2+}$ will remain in solution, separating the $Fe^{3+}$ and $Cu^{2+}$ ions.

$Cu^{2+}{}_{(aq)} + 4\ NH_{3\,(aq)} \rightleftharpoons [Cu(NH_3)_4]^{2+}{}_{(aq)}$ Eqn. (4)

$NH_{3\,(aq)} + H_2O_{(l)} \rightleftharpoons NH_4^{1+}{}_{(aq)} + OH^{1-}{}_{(aq)}$ Eqn. (5)

**Therefore the addition of $NH_3$ can be used to separate the Cu(II) cation from the iron(III) and zinc(II) cations.**

## IV. The Presence of Colored Ions

A preliminary examination of an unknown that may contain a colored cation can yield valuable information. Two of the cations for which you are testing are colored: $Fe^{3+}$ (rust or yellow), and $Cu^{2+}$ (aqua). If the unknown solution is colorless, you know immediately that these two ions are either absent or present in extremely low concentrations. However, be aware that clues can sometimes be misleading. For example, if $Fe^{3+}$ and $Cu^{2+}$ are both present, what color would you observe? Undoubtedly, the color depends on the proportions of each cation present.

## Chemicals

| Known Solutions | Testing Solutions | Confirmation Solutions | River Samples |
|---|---|---|---|
| 0.1 M $AgNO_3$ | 3 M HCl | 0.2 M NaSCN | Unknown A |
| 0.2 M $Cu(NO_3)_2$ | 6 M $NH_3$ (as $NH_4OH$) | | Unknown B |
| 0.2 M $Fe(NO_3)_3$ | 6 M NaOH | | |
| 0.2 M $Zn(NO_3)_2$ | 3 M $H_2SO_4$ | | |
| 0.1 M $Ba(NO_3)_2$ | | | |

## Equipment & Materials

| | | | |
|---|---|---|---|
| test tubes* | Parafilm squares | Plastic droppers | Centrifuge |

**Rinse and reuse the test tubes as needed.*

## Experimental Techniques

### Addition of Reagents

Always use a dropper when adding a small quantity of a liquid to a test tube. One mL is about 20 drops. If a reagent bottle has a dropper, replace it promptly after use. Do not allow the dropper to touch the container or solution to which you are adding the dropper's contents. Do not set the dropper down on the bench top or another surface, and be sure to return it to the correct bottle. Always mix thoroughly after adding chemicals. If you see layers in the test tube, the contents are not mixed well enough.

### Precipitation

To detect the formation of a precipitate on mixing two solutions, it is essential that both solutions be initially clear; if necessary, centrifuge to clarify. A "clear" solution is transparent but not necessarily colorless.

After adding a reagent to bring about precipitation, always test for complete precipitation if the purpose is to separate one substance from another. Centrifuge and add another drop of reagent to the clear supernatant. If precipitation is complete, no additional precipitate will form. However, if insufficient reagent was added the first time, the additional drop will cause formation of more precipitate. If more precipitate is observed, add several more drops of reagent, centrifuge, and again test for completeness of precipitation. Repeat until no precipitate is formed on adding reagent.

**Washing:** After the precipitate and supernatant are separated, the precipitate is washed by adding a few drops of water, mixing thoroughly with a stirring rod, centrifuging, and removing the washings with a plastic dropper. Two washings are generally necessary to prevent contamination of the precipitate. **Failure to wash precipitates is one of the most common sources of error in qualitative analysis.**

### Adjusting Acidity

Always stir well when adding acid or base. To test the pH of the solution, apply a drop onto litmus paper. *Never dip the paper into the solution*.

#### Centrifuge

The centrifuge is used to speed up the separation of a precipitate from a liquid. When a mixture of solid and liquid is placed in a tube and rotated at high speed in a centrifuge, the precipitate is forced to the bottom of the tube by a centrifugal force that is many times greater than the force of gravity.

After centrifuging, the *supernatant,* or clear liquid above the precipitate, can easily be withdrawn with a plastic dropper.

The centrifuge will be damaged if allowed to run unbalanced. Always insert an even number of test tubes in opposite positions in the centrifuge, noting their locations by number. The test tubes should contain approximately the same amount of liquid. Set the machine in motion for two minutes, which should be sufficient to achieve effective separation of the solid from the liquid. Allow the centrifuge to stop spinning before trying to remove your test tubes.

**Experimental Procedure**

**Part A: Development of an Analysis Scheme**

Use the information in the Qualitative Analysis Summary section above, along with the flow chart on the last page, to identify the cations in the known and unknown samples. Tape this in your notebook in your Calculations / Results section (3 copies; known, unknown A, unknown B).

**Part B: Separation Tests (Work with the known solution and both unknown samples.)**

1) Use the dropper bottles of known solutions to put 20 or so drops of each cation into the same, one test tube. As you are doing this, record the color of each cation solution.
2) Obtain two different unknown samples, and put approximately 3 mL of each unknown sample into its own test tube.
3) Follow the analysis scheme as you perform the following tests on the solution of known cations and each unknown sample. Record your observations for each step in your notebook. ***(Work with these three test tubes at the same time.)***

---

**THE FOLLOWING PROCEDURE STEPS APPLY TO THE KNOWN AND BOTH UNKNOWNN, WHEN APPLICABLE. If a solid does not form, then you don't have to centrifuge that particular test tube, but you still have to use that test tube for future tests.**

**To test for the silver ion: MIX WELL after each drop!!!!**

4) Add 15 drops of 3 M HCl to the test tubes containing the known cations, unknown A, and unknown B.
5) For the test tubes in which a solid formed, centrifuge the test tube to separate the solid from the liquid. Separate and save the solid and the liquid for the next steps.

***5.5) Wash the solid from step 5 after centrifuging and separating from the liquid.***

6) Add 5 drops of 6 M $NH_3$ to the solid from step 5.5 .

**To test for copper, iron, zinc, and barium ions: MIX WELL after each drop!!!!**

7) Add several drops of 6 M NaOH solution to the liquid from step 5 to make precipitate form. Add additional drops of NaOH solution until no additional solid forms. You need a basic solution at this point, but not too basic. Test the basicity with litmus paper. (Blue litmus turns red with acidic solutions; red litmus turns blue with basic solutions.)
8) Centrifuge the test tube to separate the solid from the liquid. Save the solid and the liquid for the next steps.

**To test for barium ion: MIX WELL after each drop!!!!**

9) Add 4 drops of 3 M $H_2SO_4$ to the liquid from step 8. If a precipitate does not form, test the acidity of the solution with litmus paper. Add additional drops of $H_2SO_4$ if necessary to make the solution acidic. You should see a precipitate form in the known sample.

**To test for the copper, iron, and zinc ions: MIX WELL after each drop!!!!**

10) Add 5 drops of 6 M $NH_3$ solution to the solid from step 8. Test the basicity of the solution with litmus paper. You need a basic solution. Add more $NH_3$ if necessary.
11) Centrifuge the test tube to separate the solid from the liquid. Save the solid for the next step. Observe the color of the liquid. **(The dark blue observation is for the copper ion.)**
12) Clean the solid from step 11. (WASH twice)

**To test for iron and zinc ions: MIX WELL after each drop!!!!**

13) Add 5 drops of 6 M NaOH solution to the clean solid from step 12.
14) Centrifuge the test tube to separate the solid from the liquid. Separate and save the solid and liquid for the next steps.

**To test for the zinc ion: MIX WELL after each drop!!!!**

15) In a separate test tube, mix 10 drops of 3 M HCl with 20 drops if DI water. Mix well. Use this diluted acid solution and add it dropwise to the liquid from step 14. This is your confirmation test for Zn. If zinc is present, you should see a precipitate form. If you add too much HCl, then the precipitate will dissolve or you may never see the precipitate.

**To test for the iron ion: MIX WELL after each drop!!!!**

16) Observe the color of the solid from step 14. Add 3 M HCl dropwise until the solid dissolves.
17) Add 0.2 M NaSCN dropwise as a confirmation test for $Fe^{3+}$.

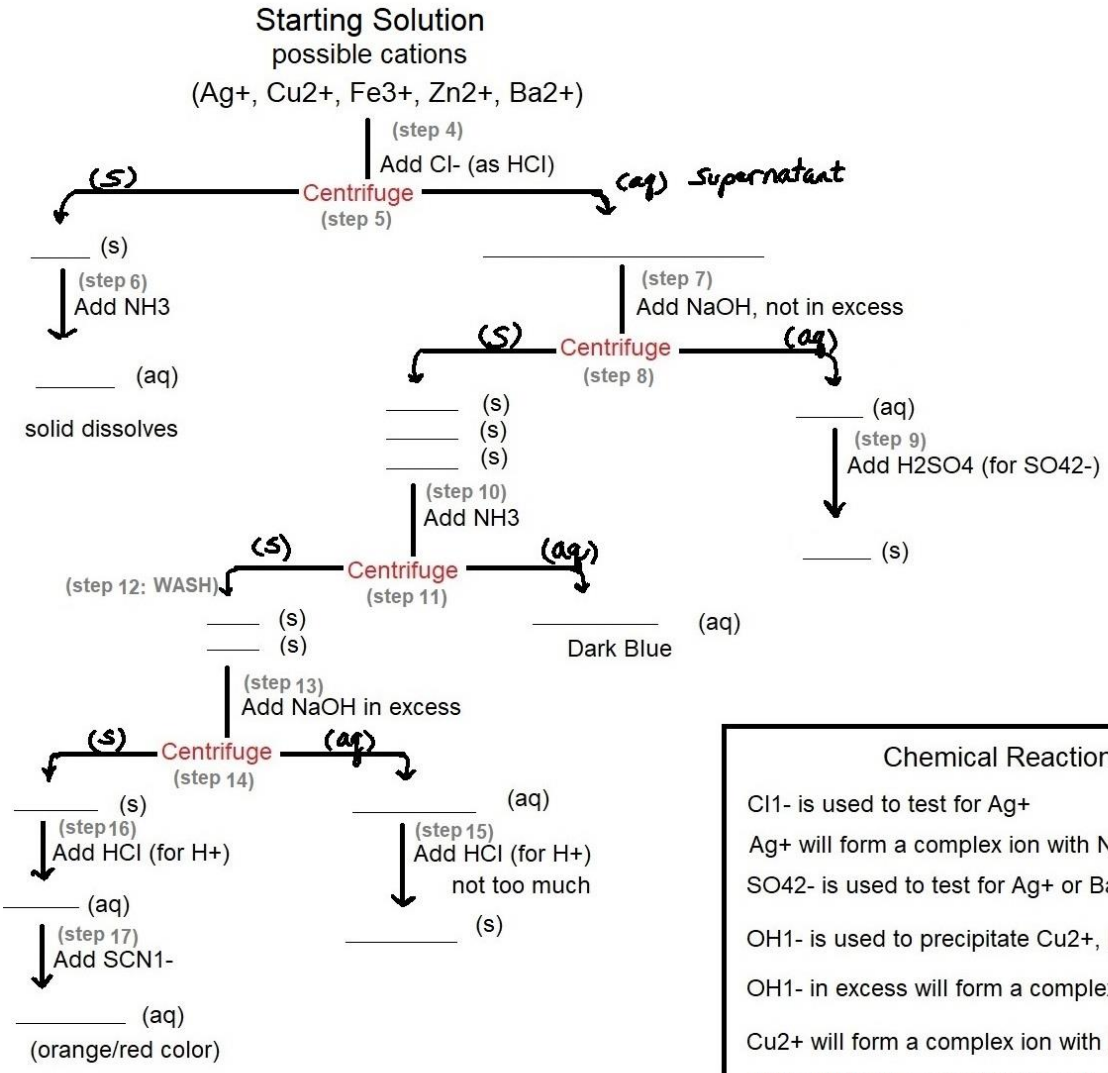
**At this point, you should be at the bottom of the analysis scheme.**
Use your observations to determine the cations present in each of your unknown samples.

For the **Data Table section** of your notebook, make a table of tests performed, observations, and cations identified

For the **Calculations / Results section** of your notebook, complete the analysis scheme with your observations, cation identifications, solids formed, and complex ions formed.

For the **Conclusion section** of your notebook, comment on the accuracy of your results for the known sample and the two unknown samples. Summarize the cations identified in each unknown.

**Analysis Scheme Flow Chart**
Please complete the chart by adding in observations, identified cations (your conclusions), and the chemical formulas of all solids and complex ions formed. *Print three copies of this chart; one for the known solution and one for each unknown. Tape all three completed charts into your notebook.*




## Chemical Reaction Facts

Cl1- is used to test for Ag+

Ag+ will form a complex ion with NH3 (aq)

SO42- is used to test for Ag+ or Ba2+

OH1- is used to precipitate Cu2+, Fe3+, Ag+, and Zn2+

OH1- in excess will form a complex ion with Zn2+

Cu2+ will form a complex ion with NH3 (dark blue)

Fe3+ will form a complex ion with SCN1- (orange/red)

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## Experiment 29: pH Measurements and Its Applications

*(This experiment was adapted from Santa Monica College.)*

**Purpose:** The measurement of pH will be practiced and applied to solution questions.

a) The pH of two different solutions will be estimated with pH paper and measured with a pH meter.
b) An unknown acid solution will be used to prepare a series of solutions by serial dilution, and the pH of each solution will be measured with a pH meter. Knowing the concentration and pH of each solution will allow for the calculation of the % ionization for each solution.
c) A buffered solution will be prepared with a weak base and its conjugate acid, $Na_2CO_3$ and $NaHCO_3$. The behavior of this buffer will be observed when a strong acid and a strong base is added. This buffer behavior will be compared to the pH changes that occur when strong acid and strong base are added to deionized water.

**Background:** The pH of a solution is a value that represents the concentration of $H_3O^{1+}$ in that solution. It is an easy-to-convey value, rather than stating concentrations in scientific notation. The most common pH scale contains values from 0 to 14. pH values less than 7 are for acidic solutions. pH values greater than 7 are for basic solutions. A pH value equal to 7 is for neutral solutions.

The pH value of a solution is calculated with the following equation:

$$\mathbf{pH = - \log [H_3O^{1+}]}$$

The pH of a solution can be estimated with pH paper; that is paper impregnated with indicators. Indicators are chemicals that have a color that depends on the pH of their environment. The value you get with pH paper is an estimated value; no significant figures.

The pH of a solution can be measured with a pH meter. The pH meter has an electrode that is sensitive to the concentration of $H_3O^{1+}$. The pH meter will report out pH values with two significant figures. Commercial buffer solutions are used to check the accuracy of the pH meter.

Once the pH of a solution is known, the concentration of $H_3O^{1+}$ can be calculated. The following equation is used for this:

$$\mathbf{[H_3O^{1+}] = 10^{-pH}}$$

A weak acid (HA) dissolved in water reacts with the water as follows:

$$HA + H_2O \rightleftharpoons A^- + H_3O^+$$

The % ionization of a weak acid is calculated with the following equation:

$$\% \text{ Ionization} = ( [H_3O^+]_{equil} / [HA]_{initial} ) \times 100$$

The pH of the solution can be used to calculate the $[H_3O^+]_{equil}$. The $[HA]_{initial}$ value can be calculated with the dilution equation.

A buffered solution is a solution that contains a weak acid and its conjugate base, or a weak base and its conjugate acid. The pH of a buffer can be calculated with the following equation:

pH = pKa + log ( [buffer base] / [buffer acid] ), and pKa = - log Ka

The pH of a buffer does not change very much when an acid or base is added to the buffer. The buffer base will neutralize incoming acid and the buffer acid will neutralize incoming base.

**Chemicals and Equipment:**

$NaCl_{(s)}$ $NaHCO_{3(s)}$ $Na_2CO_{3(s)}$ (0.10 M unknown acid) 6 M HCl
6M NaOH

pH paper pH meter pipets 100 mL vol. flasks beakers
plastic droppers Parafilm Balance, weigh paper

Ka $_{HCO3^{1-}}$ = $4.8 \times 10^{-11}$

**Procedure:**

**Part A: Prepare two different solutions, each with a concentration of 0.010 M.**

1) Obtain solid NaCl from the stock bottle.
2) Use tared weighing paper at the lab balance, and measure out the appropriate amount of solid NaCl to make 100 mL of a 0.010 M solution. Record this mass of NaCl in your notebook.
   (calculate moles and then grams, M=mole/L, mole x g/mole = g)
3) Transfer the solid NaCl from the weighing paper into a 100 mL volumetric flask.
4) Add deionized water to the flask to dissolve the solid. Then continue to add the water to dilute to the graduation mark on the neck of the flask. Make sure the bottom of the meniscus sits exactly at the calibration mark.
5) Cover the flask with Parafilm, secure with your thumb, and invert to mix well (50 inversions). Save this solution.
6) Repeat steps 1 – 5 with $Na_2CO_3$.
7) Put each of the two solutions into its own clean, dry 150 mL beaker.
8) Use a clean, dry glass stirring rod to take a drop of solution from the beaker and put it onto a piece of pH paper. Estimate the pH of each solution using the color chart supplied with the pH paper. Record these pH values in your notebook.
9) Use the pH meter to measure the pH of each solution. Record these pH values in your notebook.

**Part B: Calculation of the % Ionization for an Unknown Weak Acid**

Perform serial dilutions to prepare two solutions of different concentrations.

1) Obtain 20 mL of the 0.10 M unknown acid from the stock bottle in the fume hood.
2) Pipet 10.0 mL of this acid into a 100 mL volumetric flask.
3) Add deionized water to the flask to dilute the acid to the calibration mark. Make sure the bottom of the meniscus sits exactly at the calibration mark.
4) Cover the flask with Parafilm, secure with your thumb, and invert to mix well (invert 30 – 50 times). This is solution SD1.
5) Pipet 10.0 mL of this new solution, SD1, into a new 100 mL volumetric flask and dilute to volume, following steps 3 – 4. This is solution SD2.
6) Measure the pH of each of these solutions with the pH meter. (No pH paper)

**Part C: Preparation of a Buffered Solution**

Prepare a buffered solution (referred to as the original buffer).

1) Weigh 0.840 g of $NaHCO_3$ into a 100 mL beaker (tare the beaker).
2) Weigh 1.060 g of $Na_2CO_3$ into the same beaker (tare the beaker + $NaHCO_3$).
3) Add approximately 50 mL of deionized water to the beaker, and stir with a glass stirring rod until the solid is dissolved.
4) Rinse the glass stirring rod off with deionized water, while holding it above the beaker. This is to make sure you don't lose any of the dissolved solutes.
5) Transfer the solution into a 100 mL volumetric flask, making sure not to lose any solution. Rinse the beaker with three 10 mL portions of deionized water, transferring each rinse into the volumetric flask.
6) Dilute the solution to the graduation mark on the flask, making sure that the bottom of the meniscus sits exactly at the calibration mark.
7) Cover the flask with Parafilm, secure with your thumb, and invert to mix well (invert 50 times).

(continued on the next page)

Now the original buffer solution is prepared. Before using the buffer, divide it into two equal portions. Do this by pouring 50.0 mL of the buffer into a graduated cylinder. Then transfer each 50 mL portion of buffer into its own clean, dry 100 mL beaker (label as buffer 1 and buffer 2). You will be observing how the pH of a buffer does not change much when an acid or base is added.

8) Use the pH meter to measure the pH of one of these buffer portions (since both portions are the same, the pH will be the same for both).
9) Add 0.50 mL of 6.0 M HCl to buffer 1; mix. Measure the pH with the pH meter.
10) Add 0.50 mL of 6.0 M NaOH to buffer 2; mix. Measure the pH with the pH meter.
11) Use a new, clean, dry 100 mL beaker. Add 50.0 mL of deionized water to the beaker. Measure the pH of this water with the pH meter. The pH may not be exactly 7. If the DI water is very pure, it will be difficult to get a pH reading.
12) Add 0.50 mL of 6.0 M HCl to this water sample; mix. Measure the pH with the pH meter.
13) Use a new, clean, dry 100 mL beaker. Add 50.0 mL of deionized water to the beaker. Assume the pH is the same as in step 11.
14) Add 0.50 mL of 6.0 M NaOH to this water sample; mix. Measure the pH with the pH meter.

**Notebook**

**Part A:** In the Data Table section of your notebook, tabulate the molarity concentration of each solution, the ions present in solution, the pH obtained with pH paper, and the pH obtained with the pH meter.
In the Calculation / Results section of your notebook, mention what you expected the pH to be (pH <7, =7, >7) based on the ions in solution (were the ions weak acids, just spectators, or weak bases). Show the calculations for the mass needed for each solution.

**Part B:** In the Data Table section of your notebook, list the molarity of weak acid in each solution (this will be the Md concentrations), and the measured pH values. In the Calculation / Results section of your notebook, show the Md concentration calculation of the acid in each of these solutions (Md for SD1 and SD2). (This is the initial concentration, [HA], for the % Ionization equation.) Calculated the concentration of $H_3O^{1+}$ with $[H_3O^{1+}] = 10^{-pH}$. Calculate the % ionization for each solution. Does the weak acid ionize 100%?

**Part C:** In the Data Table section of your notebook, tabulate the pH values measured for each solution, including the water, the water with acid and water with base.

In the Calculation / Results section of your notebook, show the following:

a) Calculate the molarity of each buffer component. (g to moles, moles / L = M)
b) Calculate the expected pH of the original buffer solution.
   (pH = pKa + log([b.base]/[b. acid])
c) Calculate the Md molarity of the added HCl and added NaOH.
   (Mc*Vc=Md*Vd, solve for Md, Vc is 0.5 mL, Vd is 50.5 mL, Mc is the stock bottle molarity)
d) Write the neutralization reaction for the buffer when HCl is added.
e) Calculate the new buffer molarities after the addition of HCl (assume no dilution of the buffer components, but subtract and add for the buffer components used and made).
f) Calculate the new buffer pH after the addition of HCl by using the new molarities.
g) Write the neutralization reaction for the original buffer when NaOH is added.
h) Calculate the new buffer molarities after the addition of NaOH (assume no dilution of the buffer components, but subtract and add for the buffer components used and made).
i) Calculate the new buffer pH after the addition of NaOH by using the new molarities.

**In the conclusion section of your notebook**, comment on the results of each part of this experiment with regard to each purpose:

In Part A, you obtained pH values using pH paper and a pH meter. Compare and comment on these results. You prepared two different solutions; how did the pH values compare to what was expected, given the ions that existed in solution? Explain why.

In Part B, you prepared two solutions of a weak acid by serial dilution. How did the % ionization values compare, relative to molarity?

In Part C, you prepared a buffered solution. This was a weak base with its conjugate acid buffer. When you added the HCl and the NaOH, did the pH change much? Why or why not? Comment on the drastic change in pH of the water samples when HCl was added, and when NaOH was added, as compared to the additions to the buffer.

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## Experiment 30: Applying the Concept of Buffers

*(Part B of this experiment was adapted from Colby College, CH 142, Spring 2012.)*

**Purpose** The purpose of this experiment is to determine the Ka of a weak acid; acetic acid. In order to do this, the acid solution will be titrated with an NaOH solution, using two different methods of titration. (The NaOH solution will first need to be standardized to determine its exact concentration.)

**Background**

The NaOH solution is approximately 0.08 M, however, the exact concentration is not known. Therefore, the NaOH solution will have to be standardized. That means you will use pure potassium hydrogen phthalate (KHP) as the acid in a titration with this NaOH solution. The moles of KHP will be known and the volume of NaOH used will be known (final – initial buret volumes). You will be calculating the molarity concentration of NaOH (M = moles / Liters). The NaOH and KHP react on a 1:1 basis as follows

$$KHP_{(aq)} + NaOH_{(aq)} \rightarrow KNaP_{(aq)} + H_2O_{(l)}$$

After the NaOH solution is standardized, you will use it to titrate the acid solution. The Ka values will be determined with two different methods. The concentration of acetic acid will be determined.

**Acetic Acid:**

**Titration method 1:** The monoprotic acid will be titrated twice with the standardized NaOH solution. (This lets you calculate the concentration.) You will use the titrated solution to make a buffer solution. The buffer will be prepared by mixing the titrated solution with an equal volume of *prepared* acid solution.

When the acetic acid is titrated, the acid is used and its conjugate weak base is made. At the equivalence point, the titrated solution contains the weak conjugate base and spectator ions. When a portion of original weak acid (diluted to match the total volume of the titrated solution) is added to the titrated solution, a buffer solution is created that contains $[HA] = [A^-]$. When the concentrations of HA and $A^-$ are equal, the pH equals the pKa. You will create this buffered solution and then measure the pH. The pH value will be used to calculate the Ka value. (pH = pKa $\quad$ $pKa = 10^{-pKa}$ )

**Titration method 2:** This acetic acid will be titrated with the NaOH solution. The pH of the acid solution will be measured during the titration, after each increment of NaOH is added. An S-shaped titration curve will be made with Excel or Google Sheets so that the equivalence point and Ka can be determined.

**Chemicals**

| | |
|---|---|
| Pure potassium hydrogen phthalate (KHP), FW 204.2 g/mole | NaOH solution, approximately 0.08 M |
| Acetic acid solution, 0.050 M | |
| Phenolphthalein solution, 1% | |
| Commercial pH buffers to check the pH meter (pH = 4.00, pH = 7.00) | |

**Equipment**

| | | |
|---|---|---|
| Erlenmeyer flasks, 125 mL | Beaker, 100 mL | Beakers, assorted sizes |
| Pipet, 20 mL, 10 mL | Buret, buret stand & clamp | Balance, weigh paper |
| pH meter | Plastic droppers | Spatula, Glass stir rod |

**Procedure**

**Part A: Standardization of the NaOH Solution**

1) Use the lab balance and tared weigh paper to weigh out 0.230 g – 0.250 g of KHP. Record the exact mass and then transfer it into a 125 mL Erlenmeyer flask.
2) Add 20 mL of DI water to the flask to dissolve the KHP. Use a glass stir rod to break up any chunks of KHP, stir to dissolve, and mix well.
3) Add three drops of phenolphthalein indicator to the KHP solution in the flask; swirl to mix.
4) Set up the buret as indicated in the titration video in Blackboard:
    a. obtain the buret, buret stand and buret clamp
    b. rinse the buret with DI water
    c. rinse the buret with three, 5 mL portions of the NaOH solution
    d. fill the buret with the NaOH solution
    e. drain some of the NaOH to remove the air bubble in the buret tip
5) Record the initial volume of NaOH in the buret. All volume readings must have two digits after the decimal point, even if one or both digits are zero.
6) Place the Erlenmeyer flask with the KHP solution under the buret tip.
7) Titrate with the NaOH until the solution becomes a faint pink color. The color must linger for at least one minute. If the solution color is dark pink, you must discard the trial and do another trial.
8) Repeat steps 1 – 7 (omit step 4, the buret is already setup) until you have two usable titrations. Each trial must be done from start to finish before beginning the next trial.
9) Calculate the molarity of NaOH for each trial, and then calculate the average $M_{NaOH}$. You will use this average molarity for your calculations in parts B and C.

**Part B: Titration of the Acetic Acid Solution with titration method 1**

1) Pipet 20.0 mL of acetic acid solution into an Erlenmeyer flask.
2) Add three drops of phenolphthalein indicator into the acid; swirl to mix.
3) Refill the buret with NaOH solution if necessary.
4) Record the initial buret volume (2 digits after the decimal point).
5) Titrate the acid solution until the faint pink endpoint is reached.
6) Record the final buret volume.
7) Measure the total volume of solution in the flask with a clean, dry graduated cylinder. Record this volume in your notebook.
8) Transfer this solution back to the Erlenmeyer flask and SAVE THIS SOLUTION FOR LATER use in step 11.
9) Pipet 20.0 mL of acetic acid solution into the relatively dry graduated cylinder.
10) Add enough DI water to the cylinder to make the total volume equal to the total volume of titrated solution found in step 7. This new solution is your *prepared* acid solution.
11) Combine the *prepared* acid solution with the titrated solution in the flask from step 8. Mix well. This is your trial 1 buffered solution of acetic acid / acetate.
12) Measure the pH of this buffered solution. This pH is equal to the pKa of acetic acid.
13) Repeat steps 1 – 12 for trial 2.

**Part C: S-Shaped Titration Curve with titration method 2**

1) Pipet 20.0 mL of acetic acid solution into a clean, dry 100 mL beaker.
2) Add three drops of phenolphthalein indicator into the acid; swirl to mix.
3) Refill the buret with NaOH solution if necessary (you will need approx. 35 mL).
4) Record the initial buret volume. Start at 0.00 mL. (2 digits after the decimal point).
5) Check the pH meter with the pH = 4.00 buffer solution.
6) Put the pH electrode in the 100 mL beaker and record the pH of the acetic acid solution.
7) Start to titrate the acid solution, adding 2 mL of NaOH solution from the buret as each increment of base. After each increment, swirl to mix well and record the pH of the solution in the beaker. The pH electrode should be sitting in the beaker during the titration, off to the side so the stream of NaOH solution from the buret does not hit the electrode. Do 4 increments of NaOH addition.
8) Continue to add NaOH by 1 mL increments, mixing and measuring the pH after each increment. Do this for 2 increments.
9) Continue to add NaOH by 0.5 mL increments, mixing and measuring the pH after each increment. Do this until the pH jumps up significantly; to pH of approximately 11.
10) Continue to add NaOH by 1 mL increments, mixing and measuring the pH after each increment. Do this for 2 increments.

At this point, you have enough data to plot your S-shaped titration curve. Tabulate the total volume of NaOH solution added to the beaker at each increment and the pH of the solution in the beaker. There is a video in Blackboard to show you how to make the S-shaped titration curve.

**For the Calculations / Results section of your notebook:**

**Part A**

1) For each trial of titrated KHP, calculate the molarity of the NaOH solution.
2) Calculate the average molarity of NaOH ( (T1 + T2) / 2 ). Use this average molarity of NaOH for the calculations in Part B and Part C.

**Part B**

3) For each trial of titrated acetic acid, calculate the molarity of the acid.
4) Calculate the average molarity of acetic acid ( (T1 + T2) / 2 ).
5) Use the pH of each buffered solution of acetic acid/acetate to calculate the Ka.
6) Calculate the average the Ka value ( (T1 + T2) / 2 ).

**Part C**

7) Plot an S-shaped titration curve that shows the equivalence point for the titration of acetic acid.
8) On the graph, drop straight down from the center of the vertical pH jump to determine the volume of NaOH at the equivalence point. Divide the equivalence point volume by 2, and use this ½ volume to determine the pH that equals the pKa. Watch the video in Blackboard to see how to do this.
9) Calculate Ka for $CH_3COOH$. At the ½ equivalence point, pH = pKa, and $Ka = 10^{-pKa}$.
10) Calculate the molarity of the acetic acid solution originally used with the equivalence point NaOH volume obtained from the graph. Remember, the stoichiometry for this titration is 1 NaOH : 1 $CH_3COOH$

## Experiment 31: Oxidation Reduction Titration of Vitamin-C

*(This experiment is from Santa Monica College.)*

### Purpose

- To standardize a $KIO_3$ solution using a redox titration.
- To analyze a commercial product for vitamin-C content via titration.

### Background

The two reactions below occur in the same solution. We will use these reactions in this experiment.

(1) $KIO_3(aq) + 6\ H^+(aq) + 5\ I^-(aq) \rightarrow 3\ I_2(aq) + 3\ H_2O(l) + K^+(aq)$ *generation of $I_2$*

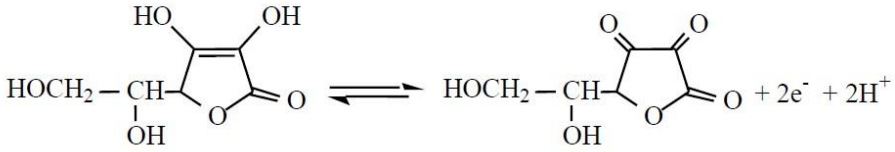
(2) $C_6H_8O_6(aq) + I_2(aq) \rightarrow C_6H_6O_6(aq) + 2\ I^-(aq) + 2\ H^+(aq)$ *oxidation of vitamin-C*

Reaction (1) generates aqueous iodine, $I_2(aq)$. This is then used to oxidize vitamin-C (ascorbic acid, $C_6H_8O_6$) in reaction (2). Both of these reactions require acidic conditions, so dilute hydrochloric acid, HCl *(aq)*, will be added to the reaction mixture. Reaction (1) also requires a source of dissolved iodide ions, $I^-(aq)$. This will be provided by adding solid potassium iodide, KI *(s)*, to the reaction mixture.

This is a redox titration. The two relevant half reactions for reaction (2) above are:

$I_2 + 2e^- \rightarrow 2\ I^-$

***Reduction half reaction for Iodine at pH 5***




***Oxidation half reaction for vitamin-C ($C_6H_8O_6$) at pH 5***

Several drops of starch solution will be added to help determine the titration endpoint. When the vitamin-C (ascorbic acid) is completely oxidized, the iodine, $I_2(aq)$, will begin to build up and will react with the iodide ions, I-*(aq)*, already present to form a highly colored $I_3$--starch complex (blue or black or grey or purple), indicating the endpoint of the titration.

### Chemicals

| | | |
|---|---|---|
| Approx. 0.01 M $KIO_{3(aq)}$ | KI solid | 1 M $HCl_{(aq)}$ |
| 1% $starch_{(aq)}$ | Ascorbic acid (pure) | Vitamin C tablet |

**Equipment**

| | | |
|---|---|---|
| 1 Buret | 1 Buret stand & clamp | 125 Erlenmeyer flask |
| Balance, Weigh paper | spatula | Beakers (several) |
| Mortar and pestle (share) | | |

**SAFETY:** Avoid contact with iodine-based solutions, as they will stain your skin. Wear safety glasses, gloves, and apron at all times during lab class.

**Proper Titration Techniques:**

**Using a Buret**

Proper use of a buret is critical to performing accurate titrations. Your instructor will demonstrate the techniques described here.

1. **Rinsing:** Always rinse a buret (including the tip) before filling it with a new solution. You should rinse the buret first with deionized water, and then three times with approximately 5-mL aliquots of the solution you will be using in the buret. Be careful to avoid spilling the solution on hands or clothing.
2. **Filling:** Mount the buret on a buret stand. Fill the buret with the titrant to just above the 0.00 mL mark.
3. **Removing Air Bubbles:** There will be an air bubble trapped in the tip of a newly filled buret. To remove the air bubble, place a waste beaker under the tip of the buret and open the stopcock fully to allow solution to flow out of the buret. This will push the air bubble out of the buret. The volume of the titrant should now be within the calibration marks on the buret.
4. **Reading the Buret:** You should always read the volume in a buret at the bottom of the meniscus viewed at eye level (see Figure 1). A black or white card held up behind the buret helps with making this reading. Burets are accurate to ±0.02 mL and all readings should be recorded to two decimal places. Be sure to record both the starting and ending volumes when performing a titration. The difference is the volume delivered.

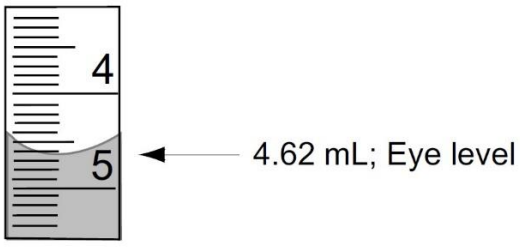



**Figure 1. Reading a Buret**

## Part 1: Standardization of your $KIO_3$ solution

The $KIO_3$ solution has an approximate concentration of about ~0.01 M. You will need to determine the exact molarity to three significant figures. You will do this by titrating pure ascorbic acid with your $KIO_3$ solution ($KIO_3$ is in the buret). Your final calculated results for each of three trials should differ by less than ± 0.005 M. Any trials outside this range should be repeated. You will need to calculate in advance how many grams of pure ascorbic acid you will need to do this standardization (this is part of your prelab exercise). Remember that your buret holds a maximum of 50.00 mL of $KIO_3$ solution and ideally you would like to use about 20 mL of $KIO_3$ solution for each titration (enough to get an accurate measurement).

*Do each trial from start to finish, before starting the next trial.*

1. Weigh approximately 0.106 g of pure ascorbic acid onto tared weighing paper. The mass you actually weigh out should be very close to 0.106 g. Record the exact mass used in each trial to three decimal places in your notebook.
2. Transfer this mass into a 125-mL Erlenmeyer flask.
3. Add 20 mL of deionized water to the Erlenmeyer flask to dissolve the solid ascorbic acid.
4. Add 0.55 g of KI, 6 mL of 1 M HCl, and 6 drops of 1% starch solution to the Erlenmeyer flask. Swirl to thoroughly mix reagents.
5. Begin your titration. As the $KIO_3$ solution is added, you will see a blue or black or grey color at the place were titrant hits the solution in the flask. While adding the $KIO_3$ swirl the flask to remove the color. As you get closer to the endpoint, the color will linger. Add the $KIO_3$ slowly as you approach the endpoint. Rinse the sides of the flask with DI water periodically during the titration. Stop adding the $KIO_3$ when the color does not disappear.
6. Calculate the molarity of this sample. Repeat the procedure until you have three trials where the molarities differ by less than ± 0.005 M. However, don't do more than 4 trials.

## Part 2: Analysis of the Vitamin-C Tablet (The Unknown)

1. Obtain one vitamin-C tablet.
2. Weigh the tablet and record its mass in your notebook.
3. Grind the tablet into a fine powder using a mortar and pestle.
4. Weigh out approximately 0.150 grams of the powdered tablet using tared weighing paper. Record the exact mass used in your notebook.
5. Transfer all of the sample from the weigh-paper into a 125 mL Erlenmeyer flask.
5. Dissolve the sample in 20 mL of deionized water and swirl well. Note that not all of the tablet will dissolve as commercial vitamin pills use insoluble binders to form the tablet.
6. Add 0.55 g of KI, 6 mL of 1 M HCl, and 6 drops of 1% starch solution to the flask before beginning your titration. Swirl to mix.
7. Begin your titration. As the $KIO_3$ solution is added, you will see a blue or black or grey or yellow color at the place were titrant hits the solution in the flask. While adding the $KIO_3$ swirl the flask to remove the color. As you get closer to the endpoint, the color will linger. Add the $KIO_3$ slowly as you approach the endpoint. Rinse the sides of the flask with DI water periodically during the titration. Stop adding the $KIO_3$ when the color does not disappear.
8. Perform two more trials. If the first titration required less than 17 mL of $KIO_3$, increase the mass of unknown slightly in the remaining trials. Make sure you have three usable trials before ending your experiment. A usable trial is one in which you did not titrate past the endpoint.

**Calculations/Results section of your notebook:**

**Part 1**

Calculate the molarity of the $KIO_3$ for each trial.
Determine the average molarity of the $KIO_3$ solution ( $(M_{T1} + M_{T2} + M_{T3}) / 3$ ) and use this molarity for calculations in Part 2.

**Part 2**

a) Use the average $M_{KIO3}$ to calculate the moles of $KIO_3$ used in each trial.
b) Use the moles of $KIO_3$ from calc. (a) to calculate the moles of Vitamin C in each trial.
c) Calculate the grams of Vitamin C determined in each trial; convert to mg.
d) Calculate the milligrams of Vitamin C per tablet, then calculate the average.

$$\text{mg Vit. C in a tablet} = \frac{\text{mg Vit. C in powder used}}{\text{g of powder used}} \times \frac{\text{g tablet}}{\text{1 tablet}}$$

e) How does your mg / tablet Vitamin C average compare to the amount listed on the bottle label? Calculate the % error to use in the conclusion. A % error less than 5% is good.

## Experiment 32: Independent Project – Method Development

### Purpose
The purpose of this experiment is to develop an acid base titration experiment for the analysis of an unknown acid. This will include testing three different indicators and selecting the best volume to use for the acid solution being analyzed.

### Background
An acid base titration relies on an acid base neutralization reaction. The equivalence point is when the moles of $H^+$ equals the moles of $OH^-$. Since the equivalence point cannot be seen visually, an indicator must be used. The color of the indicator depends on the pH of its surrounding. The change in color of the indicator, referred to as the endpoint, will be used to indicate the equivalence point.

The selection of the indicator is critical. The endpoint must be at the same point of the titration as the equivalence point. Two different indicators will be tested, to determine which one is best for the combination of acid and base used for this titration method. To make sure the indicator color change matches with the equivalence point, an S-shaped titration curve will be made. The large pH increase during the titration will show the equivalence point. The volume of titrant added, and the pH and color of the solution in the receiving beaker will all be recorded.

The volume of acid to use for each trial is important. Typically, three trials of the titration are done during an acid base titration experiment. The buret should only have to be filled once to perform all three trials. Therefore, a trial should not use more than 15 mL of titrant (the base solution). To get good significant figures, a trial should use at least 10 mL of titrant. Determine the best volume of acid to pipet into the Erlenmeyer flask so each trial will use 10 mL – 15 mL of titrant.

### Chemicals
Acid: unknown, assume monoprotic, a solution of approximately 0.050 M – 0.075 M
Base: 0.10 M NaOH solution
Indicators (in solution form): methyl orange, phenolphthalein, bromothymol blue

### Equipment
Buret, 50 mL, buret stand, buret clamp, funnel
Beaker, 100 mL – 150 mL, as the receiving vessel during the titration
Graduated cylinder, 10 mL
Erlenmeyer flask, 125 mL
Beakers, medium size, for obtaining the acid and the base solutions
pH meter for measuring the pH during the titration

## Experimental Procedure

### Part A: Determining the best indicator to use for the titration

For each of the three indicators, do a titration with the NaOH solution as the titrant in the buret. Use 20 mL of acid, measured with a graduated cylinder, in the receiving beaker. Add three drops of indicator to the acid solution in the beaker. Perform a titration as you did in Experiment 30, when you had to make an S-shaped titration curve. Record the volume of NaOH used and the color and pH of the solution in the receiving beaker at each increment of NaOH added. Plan the increments of NaOH to get enough data points to determine where the end point occurs and where the equivalence point occurs, relative to the volume of NaOH added to the beaker. You will **not** be making an S-shaped curve; you will be using your raw-data tables when determining the best indicator.

### Part B: Determining the best volume of acid to use (calculations only)

For a titration of this acid with the 0.10 M NaOH, the volume of NaOH used for each trial should be between 10 mL and 15 mL. You are using a graduated cylinder for this method development, but for an actual experiment, a student would be pipetting the acid solution into an Erlenmeyer flask. Consider the volume of base used to reach the equivalence point (the large increase in pH) for the titration in Part A, and an easy volume of acid to pipet. How much acid should be used for each trial? This section of the project, Part B, involves calculations. No additional experimentation is needed.

### Part C: Performing three trials of your titration method

Use the best indicator chosen in Part A with the volume of acid determined in Part B. Perform three trials of the titration. You do not have to measure the pH of the solution in Part C. You are just doing three trials of the titration and using the data to calculate the molarity of the unknown acid solution for each trial, and then the average molarity of the acid.

### Notebook:

Raw Data: Record the data obtained for Part A and Part C. Show your calculation for Part B.

Experimental Procedure: Write the experimental procedure that a student would have to follow to complete three trials of the titration based on your method (just a titration without a pH meter). This is what you did for Part C.

Data Tables: Make a table of expected students results (your results from Part C; volume of acid used, indicator used, initial and final volumes NaOH for each trial).

Calculation/Results: Part A: For each indicator, make a table to show the volume of NaOH added to the beaker at the endpoint, the color at the endpoint, and volume of NaOH added at the equivalence point. Part C: Show the calculation of the acid molarity in Part C for each trial and calculate the average molarity of the acid.

For your Conclusion section, state the best indicator to use and the best volume of acid to use. Explain your logic for choosing the indicator. Explain your logic for choosing the volume of acid to use. State the determined average molarity of the unknown acid solution from Part C.

## Experiment 33: Electrochemistry and Organic Molecules

*(The notebook rubric will not be followed for this experiment. Please follow the notebook directions listed in this experiment. The points for each section are listed; there is a total of 54 points available, plus 5 bonus points.)*

### Purpose

There are two separate, mini experiments for Experiment 33.
The Electrochemistry mini experiment will require a voltaic cell to be constructed and the voltage of the cell to be measured.
The Organic Molecules mini experiment will require the building of 5 molecular models, with questions based on the models.

### Background

Electrochemistry: The voltaic cell uses a spontaneous oxidation reduction (redox) reaction. Each component of a voltaic cell has a purpose, and these components are: anode, cathode, wire, salt bridge, and strong electrolyte solutions. When identifying the oxidation and reduction reactions for the two elements involved, the element with the more positive reduction potential is reduced at the cathode.

One voltaic cell will be constructed using a zinc electrode in its zinc ion solution as one half of the voltaic cell. The other half of the voltaic cell will be a graphite electrode in a hydrochloric acid solution. The graphite electrode is not part of the chemistry; it is simply a surface at which electrons are transferred.

### Chemicals

Electrodes: Graphite, Zinc
Electrolyte Solutions: (each 0.1 M) HCl, $Zn(NO_3)_2$
Saturated solution of NaCl for the salt bridge

### Equipment

Beakers (150 mL), two, to use for half reactions
Electrical wires (2) with alligator clips
Graduated cylinder
Voltmeter (1)
Tubing and cotton ball for the salt bridge
Beakers, medium size for obtaining solutions
Plastic droppers

Molecular modeling kit

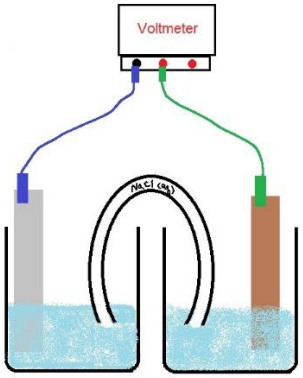
## Experimental Procedure

### Construction of the Voltaic Cell

1) Obtain two beakers (150 mL), two wires, one voltmeter, one piece of tubing, one cotton balls, one graphite electrode, and one Zn electrode. Obtain approximately 100 mL each of the electrolyte solutions: $Zn(NO_3)_2$, HCl.

2) Build the following voltaic cell by following the setup shown below:
   Zn electrode in the $Zn(NO_3)_{2(aq)}$ and the graphite electrode in the $HCl_{(aq)}$

   Record your observation and measured voltage. Calculate the expected voltage of the cell using standard state reduction voltages.




### For Your Worksheet

1. Draw your voltaic cell and label all of the components. **(6 points)**
2. Briefly write what each component is for. **(6 points)**
3. Write the half reactions. One half reaction will be oxidation and the other half reaction will be reduction. Show the electrons in each half reaction. **(2 points each)**
4. Write the whole redox reaction. Make sure it is balanced. **(5 points)**
5. Calculate the expected voltage of the cell. **(5 points)**
6. State the measured voltage and compare it to the expected voltage. **(4 points)**
7. Optional: do the extra credit calculation described below.

### Extra Credit Calculation

For the voltaic cell, use your measured voltage to calculate the ΔG for that cell.
The equation that relates ΔG to the voltage of the cell is:

$$\Delta G = - nFE_{cell}$$

n = the number of moles of electrons transferred in the balanced redox reaction. The value of n will be a whole number (1, 2, 3, 4 etc…).

F is Faraday's constant 96,485. J / (v·mole), $E_{cell}$ is the measured voltage.

This calculation is worth 5 points of extra credit for Experiment 33.

**Organic Molecules**

The IUPAC system of naming is used for this experiment. The following information is needed to build your models, to identify the bonds within the molecules, and to draw the structures.

**Table 1: Base Part of Name (the Number of Carbons in the Main Chain)**

| Number of C Atoms | Base Part of Name | | Number of C Atoms | Base Part of Name |
|---|---|---|---|---|
| 1 | meth | | 6 | hex |
| 2 | eth | | 7 | hept |
| 3 | prop | | 8 | oct |
| 4 | but | | 9 | non |
| 5 | pent | | 10 | dec |

**Table 2: Ending Part of Name (Bonding)**

| Type of Bonding in between C Atoms | Ending Part of Name |
|---|---|
| All single bonds | ane |
| At least one double bond | ene |
| At least one triple bond | yne |

If the molecule is a ring, use "cyclo" in the name.
If there are branches in the molecule, each branch will need an address number.
If there is a double or triple bond in the molecule, it will need an address number (use the lower address number of the two carbons involved with the double or triple bond).

**Table 3: Electron Domain Geometry and Hybridization**

| Number of Electron Domains around the 'Central' Atom | Electron Domain Geometry | Ideal (Predicted) Bond Angle | Hybridization |
|---|---|---|---|
| 2 | Linear | 180° | sp |
| 3 | Trigonal Planar | 120° | $sp^2$ |
| 4 | Tetrahedral | 109.5° | $sp^3$ |

**Molecules to Build:** Build a molecule for each of the following, and provide your answers in your worksheet:

1) Ethane:
Show the instructor your model (1 point)
Molecular formula (1 point)
Expanded structural formula (1 point)
Condensed structural formula (1 point)
Which orbitals are overlapping to make the C-C bond? (1 point)

2) 1-Propene:
Show the instructor your model (1 point)
Molecular formula (1 point)
Expanded structural formula (1 point)
Condensed structural formula (1 point)
What is the hybridization of each carbon atom? (1 point)

3) 2-Butyne:
Show the instructor your model (1 point)
Molecular formula (1 point)
Expanded structural formula (1 point)
Condensed structural formula (1 point)
Which orbitals are overlapping to make the triple bond? (answer for both the sigma and pi overlaps) (1 point)

4) Cyclobutane:
Show the instructor your model (1 points)
Molecular formula (1 point)
Expanded structural formula (1 point)
Condensed structural formula (1 point)
Which orbitals are overlapping to make the C-H bonds? (1 point)

5) 1-Ethyl Cyclopentane
Show the instructor your model (1 point)
Molecular formula (1 point)
Expanded structural formula (1 point)
Condensed structural formula (1 point)
What is the hybridization for each carbon atom in the ring? (1 point)

**Name:** _____

**CHE118 Experiment 33**

(Worksheet to hand-in for grading instead of your notebook)

**Voltaic Cell**

1. (6 points) Draw your voltaic cell and label all of the components

2. (6 points) Briefly describe what each component is for.

| | |
|---|---|
| Anode | |
| Cathode | |
| Salt Bridge | |
| Wires | |
| Electrolyte Solutions | |
| Volt meter | |

3. (2 points each) Write the half reactions. One will be for oxidation, one will be for reduction. Show the electrons in each half reaction.

4. (5 points) Write the whole redox reaction. Make sure it is balanced.

5. (4 points) Calculate the expected voltage of the cell.

6. (4 points) State the measured voltage and compare it to the expected voltage.

7. (5 points) Extra Credit Calculation:

Organic Molecules

1) Ethane:

Show the instructor your model (1 point)

Molecular formula (1 point)

Expanded structural formula (1 point)

Condensed structural formula (1 point)

Which orbitals are overlapping to make the C-C bond? (1 point)

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2) 1-Propene:

Show the instructor your model (1 point)

Molecular formula (1 point)

Expanded structural formula (1 point)

Condensed structural formula (1 point)

What is the hybridization of each carbon atom? (1 point)

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3) 2-Butyne:

Show the instructor your model (1 point)

Molecular formula (1 point)

Expanded structural formula (1 point)

Condensed structural formula (1 point)

Which orbitals are overlapping to make the triple bond? (answer for both the sigma and pi overlaps) (1 point)

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4) Cyclobutane:

Show the instructor your model (1 points)

Molecular formula (1 point)

Expanded structural formula (1 point)

Condensed structural formula (1 point)

Which orbitals are overlapping to make the C-H bonds? (1 point)

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5) 1-Ethyl Cyclopentane

Show the instructor your model (1 point)

Molecular formula (1 point)

Expanded structural formula (1 point)

Condensed structural formula (1 point)

What is the hybridization for each carbon atom in the ring? (1 point)

**Review Questions for the Lab Practical Exam**
(This is only a partial review. The answers are on the following page.)

1) The equation of a best-fit-line is given as Y = 87.6 X + 0.0003
The absorbance of an unknown solution is measured as 0.342
What is the concentration of the unknown solution?

2) The pH of a solution is measured as 4.62 . What is the $[H_3O^{1+}]$?

The pH of a 0.030 M an acid solution is measured as 3.20 . What is the % ionization of this acid?

What is the pH of a buffer solution consisting of 0.26 M HA and 0.24 M $A^{1-}$? The Ka for HA is $1.8 \times 10^{-6}$ .

If 0.03 M of strong base is added to the buffer in the question above, what will the new molarities of the HA and $A^{1-}$ be?

3) What is the concentration of the acid solution that required 15.80 mL of 0.13 M NaOH to reach the end point (equivalence point) in a titration? The volume of acid used was 20.0 mL.

What is the volume of base, 0.13M NaOH, needed to neutralize 20.0 mL of 0.12 M $HNO_3$?

Write the neutralization equation for the reaction of $HNO_3$ and KOH.

5) Miscellaneous questions:

What is the concentration of $CuCl_2$ when 2.0 mL of a $1.0 \times 10^{-2}$ M solution is diluted to 50.0 mL?

How much $CuCl_2$ stock solution do you need to make 100.0 mL of a $5.0 \times 10^{-3}$ M solution? The stock solution is $2.0 \times 10^{-1}$ M.

If you had 1.062 g of $CuCl_2$, how many moles of $CuCl_2$ would you have?

If you had 0.065 moles of $CuCl_2$, how many grams of $CuCl_2$ would you have?

If you had 0.065 moles of $CuCl_2$, how many moles of $Cl^{1-}$ would you have?

Answers:

1) 0.0039 (units not given in question)

2) $[H_3O^{1+}] = 2.40 \times 10^{-5}$ M
2.1 % ionization
pH = 5.71
HA = 0.023 M $A^{1-} = 0.027$ M

3) Molarity of the acid = 0.10 M
Volume of the base = 18 mL
$HNO_3 + KOH \rightarrow H_2O + KNO_3$

5) Diluted, new concentration = $4.0 \times 10^{-4}$ M
Volume of the concentrated solution needed = 2.5 mL
0.007899 moles of copper(II) chloride
8.7 g of copper(II) chloride
0.13 moles of $Cl^{1-}$