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# Creating Graphs Using Microsoft Excel and Word

*Created by Carlos Gonzalez, 2006*

## Starting Out

Microsoft Office has a built in feature to create graphs from a data table. The following information will help you create different types of graphs, make easy analysis of graphs, and make them easy to read and display information.

No matter what program in Microsoft Office you are using (Word, Excel, or PowerPoint); the graph function will be available.

- *To create a new graph in Word: select Insert → Picture → Chart.*
- *To create a new graph in Excel or PowerPoint:* select *Insert → Chart.*

## Choosing the Type of Graph

After selecting chart in PowerPoint or Word, a graph and datasheet will be displayed.

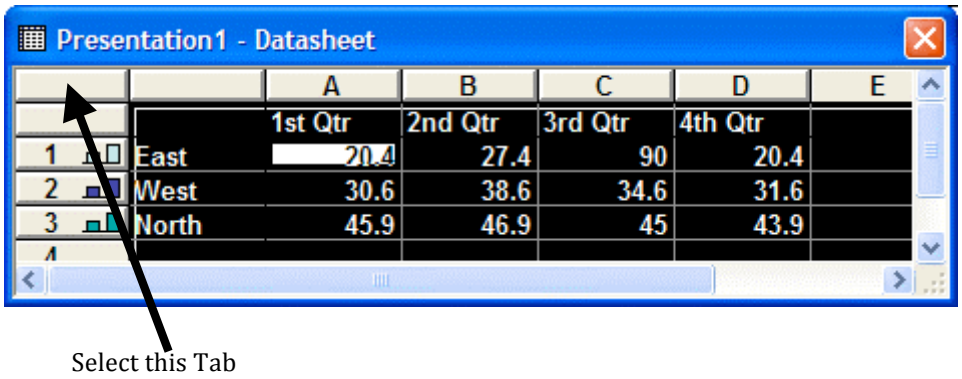
** Using this part with Excel will be explained later.*

When the chart area is displayed, right-click on the inside area (but not on the chart) and select *Chart Type*.

The window will have two tabs, "Standard Types" and "Custom Types". Make sure "Standard Types" is the tab you selected. In the list below, select the graph that best fits your needs. Then choose how you want it to look in the Chart Sub-Type and then click OK.

You will now notice that the sample graph has been changed to the type of graph you selected.

The datasheet should still be open at this point, right click on the very top left tab:

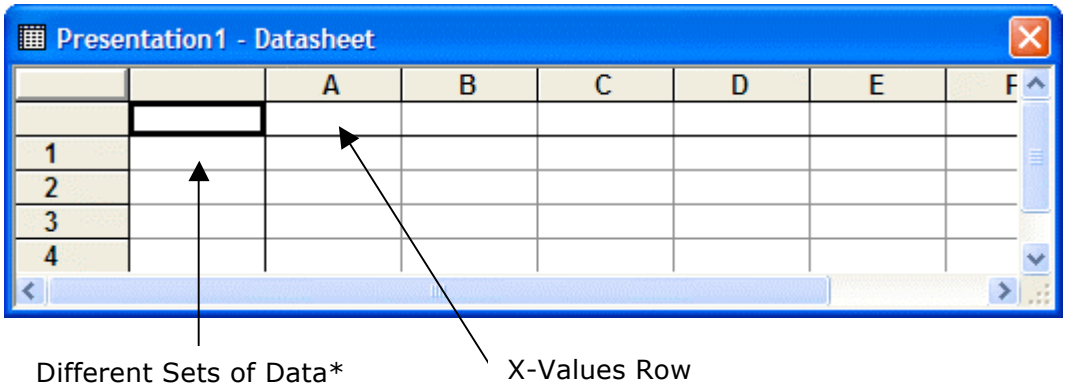



Select this Tab

You will notice it will select everything. Press the Delete Key to erase the entire table. This will make the chart in the chart area disappear. DO NOT PANIC! There is no data available to make a chart; this will be explained next. This page will help you with the two simplest types of graphs: bar and line graphs.

**Bar Graphs**

If you selected to make a bar graph, your data ***must*** be in the following format on the datasheet.




Ok, "Different Sets of Data" indicate how many bars you want. For example, if you have multiple trials of data, you can put "Trial 1" in Row 1 and "Trial 2" in Row 2. This would create multiple bars in your graph.

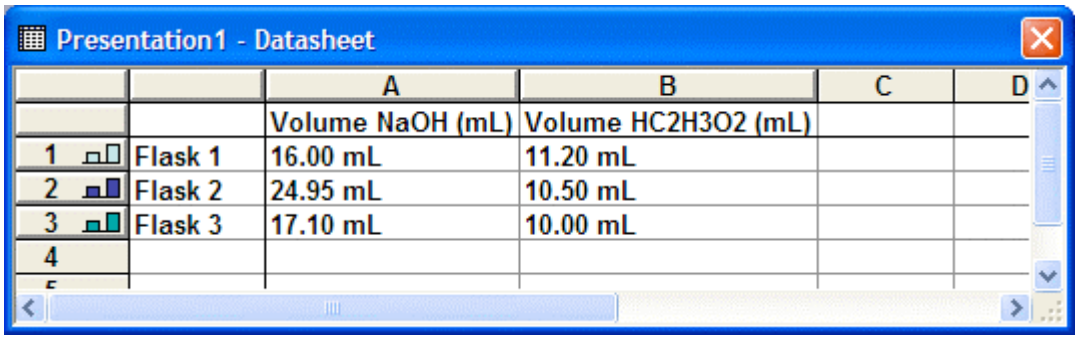
Your X-Values are going to start on Column A and keep going along that row.

The rest of the data will go on the row where a "Set of Data" is defined. These will be your Y values. Here is a sample data table and below will show you the proper orientation in the datasheet.

**Data from Titration Lab**

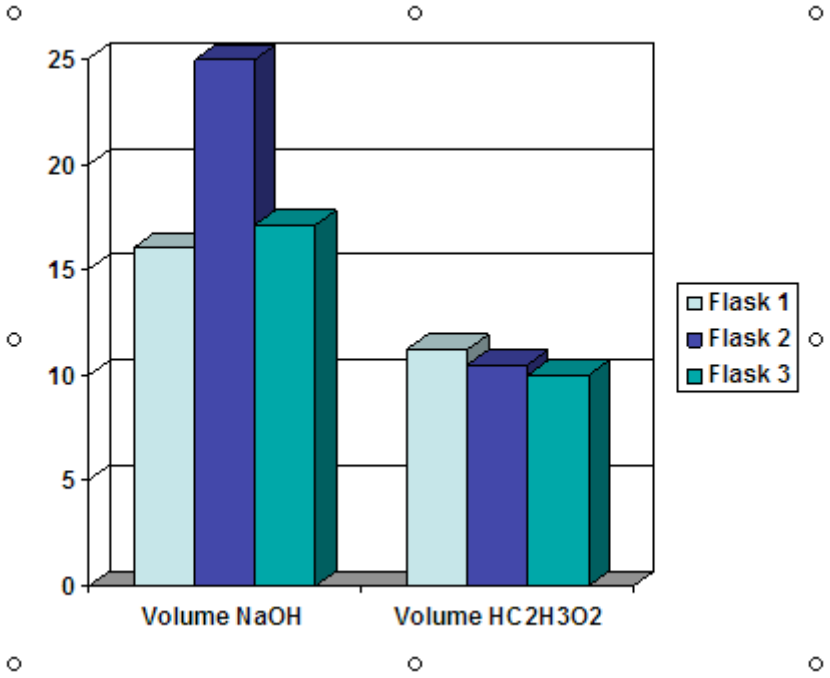
| **Flask** | **Volume NaOH (mL)** | **Volume $HC_2H_3O_2$ (mL)** |
|---|---|---|
| 1 | 16.00 | 11.20 |
| 2 | 24.95 | 10.50 |
| 3 | 17.10 | 10.00 |

Datasheet:




(This will give a graph on the following page.)

Graph:




The graph will then look like that above. However, it is missing some labels that will be discussed later. Now for a line graph…

**Line Graphs**

Line graphs are very similar to bar graphs by the data sheet; however, the data will be displayed differently. Line graphs also have additional features that bar graphs do not. Also the type of line graph you select will affect your graph's look. On the chart type selection choose Line if you want the data already connected in a linear progression. If you have data that is just a bunch of points and you want to find the best-fit line, select the XY (Scatter) option and select just the point values.

The data sheet will work the same was as the bar graph.

Here is some sample data, the datasheet, and graph:

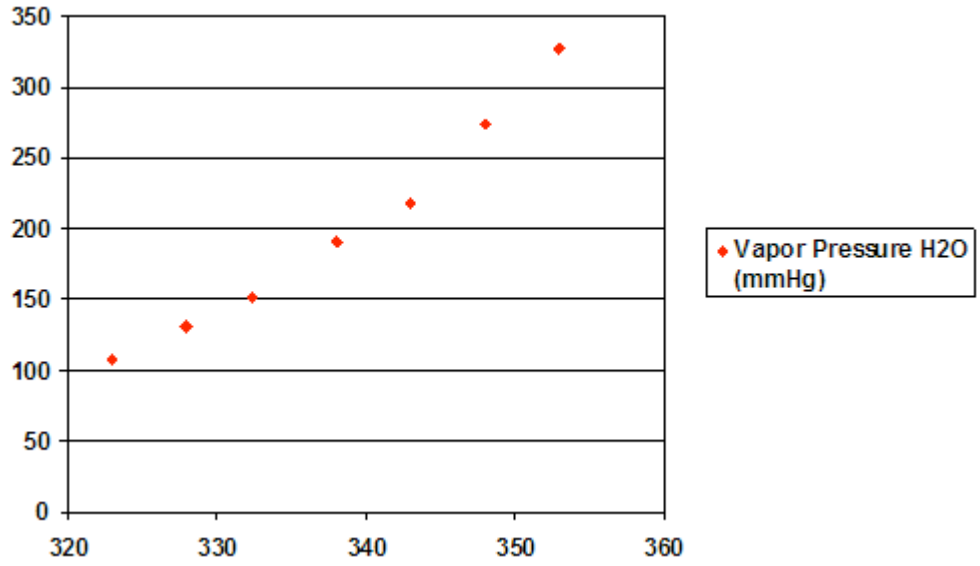
| **Temperature (°K)** | **Vapor Pressure $H_2O$ (mmHg)** |
|---|---|
| 353.0 | 326.952 |
| 348.0 | 273.904 |
| 343.0 | 217.284 |
| 338.0 | 190.912 |
| 332.4 | 151.316 |
| 327.9 | 131.480 |
| 323.0 | 107.312 |

Datasheet:

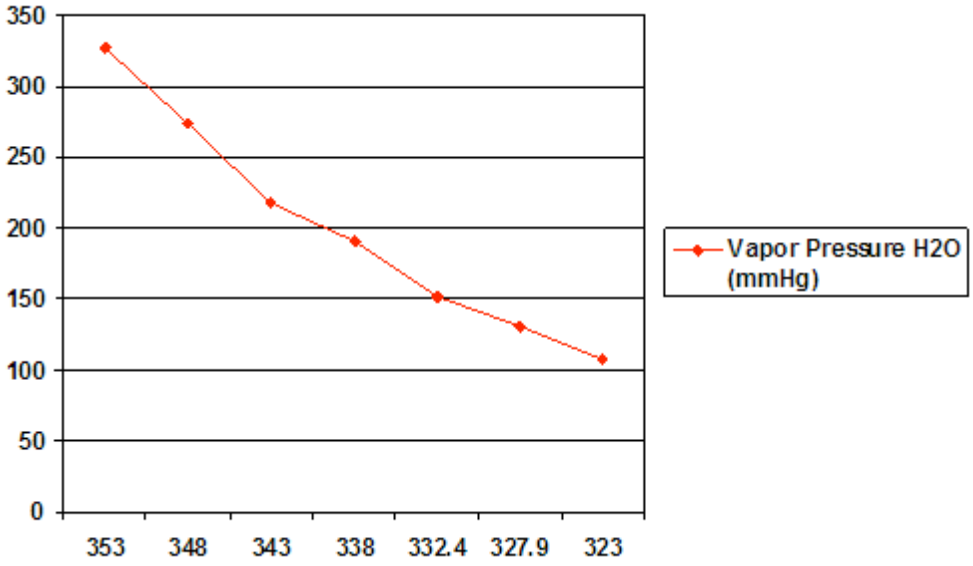
Presentation1 - Datasheet

| | | A | B | C | D | E | F | G | H |
|---|---|---|---|---|---|---|---|---|---|
| | Temperature (°K) | 353 | 348 | 343 | 338 | 332.4 | 327.9 | 323 | |
| 1 | Vapor Pressure H | 326.95 | 273.9 | 217.284 | 190.912 | 151.316 | 131.48 | 107.312 | |
| 2 | | | | | | | | | |
| 3 | | | | | | | | | |

Graph:
(XY Scatter)




(Line)



If you look at the two graphs, the XY Scatter is just like a coordinate plane where the Line graph is not. The Line graph is merely a line that shows a trend in a series of data. The XY Scatter is what you will need to use for most graphs in this course.

**Best Fit Lines in XY Scatter**

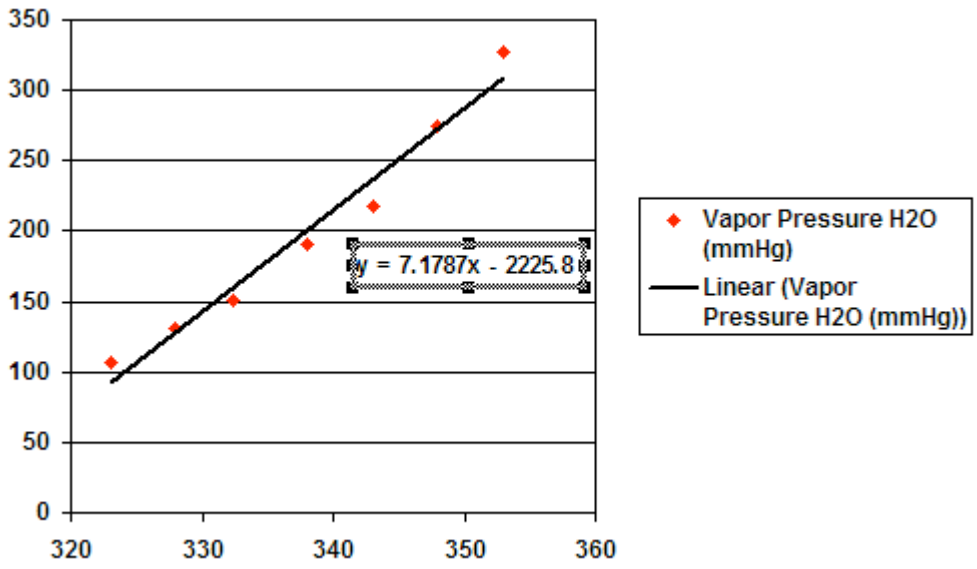
A best-fit line would be great to show an average of the data in a linear method, or it can be used to attain an equation of a line.

A best-fit line can be achieved by the following step. *(You must have the chart area open for editing.)*

Select *Chart* → *Add Trendline…*

A window will then appear that gives the type of trend line you can insert. Depending on the data you have, you may want to select a different type. Since I want the linear best-fit line, I will select a linear best-fit line. Note that you might need to use a different type of line for different sets of data. Trying out multiple fits never hurts. On the Options Tab, you can put a name on the line (best if you have multiple best-fit lines), and adjust some of its properties. The line can be extended in both directions (helpful if you predict something). The one option that will help you the most is to select the "Display Equation on Chart" option. Select it and press OK.

The chart will then look like this:




You can use the $y = mx + b$ equation to then substitute values, determine the slope, and predict values.

**Chart Labels and Formatting**

Ok, so you now have all your data plugged into a graph (bar or line); however nothing is labeled and your data might not be displayed the best way. Right-click outside the chart in the chart area and click on Chart Options.

This window has many tabs that are mostly self-explanatory, yet I will go through a few of them.

- The titles tab can be filled in with the proper labels and it will give you a preview of how your graph will look.
- The axes tab needs to have the values on them… do not change this.
- The gridlines tab is for the lines going across your graph. If you like the look of graph paper select both x and y. If you like no lines, then de-select everything.
- The legend tab basically asks where you want the legend to be displayed if you choose to display it (recommended for multiple lines/bars).
- The data labels tab can be handy if you want the y-values displayed on the graph.

Once all that is set up, click OK. The graph should now be set up and labeled properly.

If you wish to change the way the graph looks (font and size) right-click on chart area and click on "Format Chart Area." This will format the entire chart, all titles, and any labels on the graph. Each one can be selected and changed individually if you wish to change them again.

**Editing the X and Y Axes**

You may wish to have different values on your axes than they are given. Right-click on the axis you wish to edit and you will see that the only tab you want to edit would be the scale tab.

The chart is automatically sized to best fit your data; however, you may want to change it to best fit your trend line. Inserting a value will automatically change it, and it will not automatically adjust until you click the check mark next to it.

Click OK when you have made the proper changes.

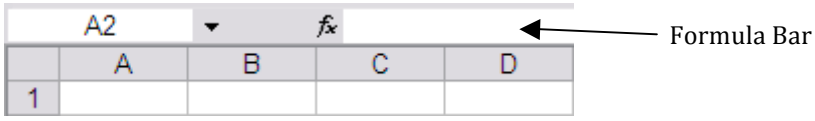
**Finished!**

This completes how to create a graph. There are a few things I have left out about changing things. However, you can right-click on almost every object and change almost anything about it. Feel free to play with the graph until you get it to how you like it!

***Hints and Formulas with Excel**

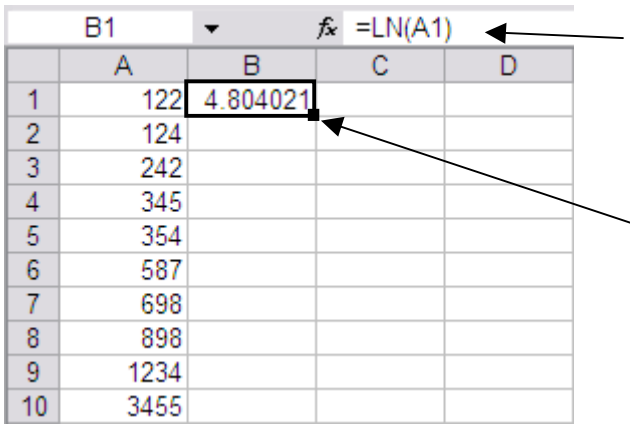
Excel also has some help in calculating mass amounts of data. This will save you a lot of time in some labs where there may be 6 trials that involve multiple calculations. The formula bar (found directly above the spreadsheet area) can help you do simple to complex calculations very quickly and easily.

The formula bar works like a calculator. Knowing how to use this feature will greatly help you in calculations. Here are a few sample formulas with simple calculations:

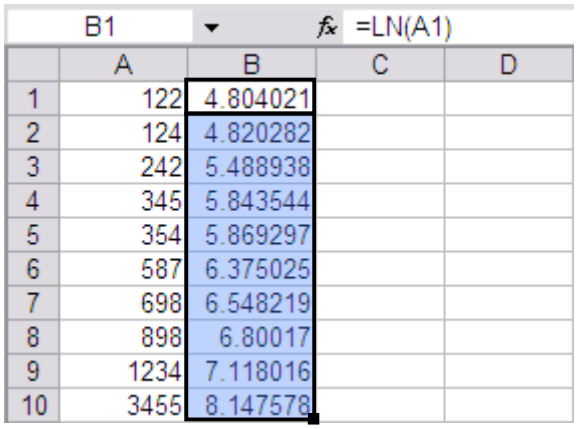



Ok, in this screen you can see that cell C1 has the formula "=A1+B1". You can easily see that it adds the two cells, A1 and B1, together.

Now, for larger sets of data, you can drag your formula across multiple cells. Let's say that you have 10 cells of numbers and you want to take the natural log of them all. Instead of working them all out on the calculator, have Excel just give you the answers. Here's how it works.

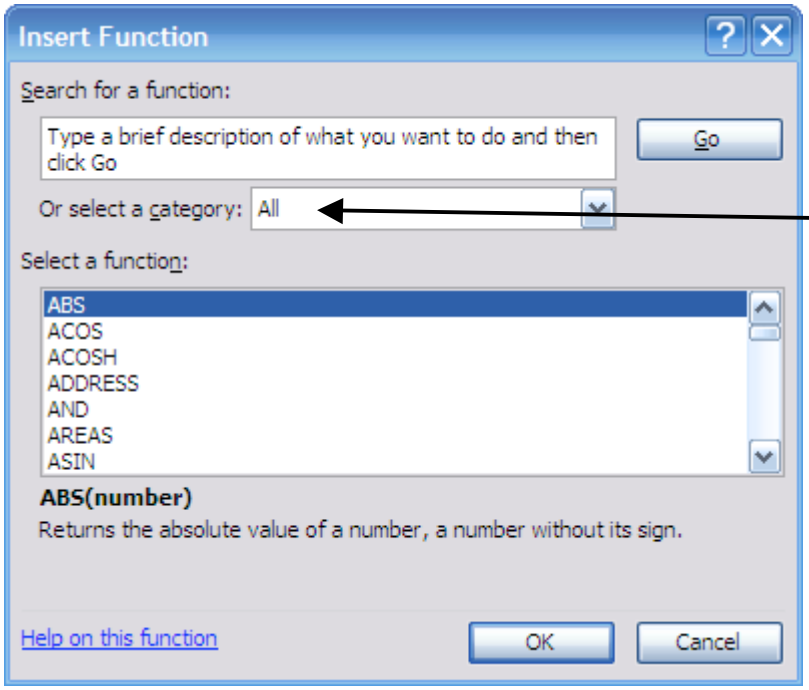



Then you get…




* You can always copy and paste your data to Word. The boarders will not show up though, so be sure to change that!

There are a lot of different formulas that you can use. If you go to *Insert* → *Formula* there is a rather comprehensive list of formulas you can choose. It will also help you with the syntax of the formula. You can never use too many parenthesis, I can't stress this enough.




Make sure this says "All" for a complete list.

Once you have all the data you need, organize what you want to graph and start creating your graph by selecting data.

Keep in mind that Excel is the easiest program to graph with, however, Word works perfectly fine as well.

**Wrap Up**

Hopefully this guide has helped you to make professional looking graphs and enhance the quality of your lab reports. It never hurts to experiment with your data and see what you can come up with!

# SEPARATION & PURIFICATION

Duration: 1 day

**INTRODUCTION**

The four major steps involved in the analysis of a substance are 1) collection of the sample, 2) preparation for measurement, 3) measurement, and 4) evaluation of data. This experiment is designed to illustrate four techniques commonly used to purify and otherwise prepare a sample for measurement. These are decantation, filtration, coagulation, and distillation. A brief discussion of these techniques is presented below. The remaining steps are subjects of future experiments and discussions.

Decantation

Many liquids can be easily separated from unmixed solids by decantation or carefully pouring the liquid off. An example would be a mixture of sand and water. The decantation process is illustrated in Figure 1.1.

Filtration

A solution quite often contains solid matter whose density is almost the same as the density of the liquid portion. When this is the case, the solids do not settle to the bottom, and separation by decantation is impossible. Under this circumstance, filtration is useful. Filters are composed of insoluble solids whose pore size allows the liquid portion of a sample to pass through but not the solid portion. Quite often it is the solid portion that is desired for analysis. In either event, a separation is effected. Folding of the filter paper is illustrated in Figure 1.2 and a typical apparatus for gravity filtration is shown in Figure 1.3.

Coagulation

A solution may contain suspended particles of colloidal size that pass through filter paper. However, they can be forced to settle by the formation of a gelatinous precipitate in the solution, which as it falls to the bottom of the container, carries down most of the insoluble matter. This process is known as coagulation and is used in many municipal water treatment plants as one of several purification steps.

Distillation

Distillation is a common purification technique that is used in the separation of volatile liquids from undesirable impurities that are usually non-volatile. A typical experimental distillation apparatus is shown in Figure 1.4. The volatile liquid to be purified is vaporized in the round-bottom flask, converted back to a liquid in the condenser, and finally collected in the receiving flask. The non-volatile impurity remains in the original mixture. To avoid bumping (when air trapped in a bubble rapidly escapes and just makes a frightful mess!), especially when solid material is present in the bottom of the distillation flask, 2-3 small boiling chips, or $CaCO_3$ pellets, are added to the flask before heating.

**OBJECTIVES**

In this experiment, you will

- Separate a mixture using four techniques.
- Test for the purity of the sample.

**MATERIALS**

Mixture of sand, dirt, salt, and water
Beakers
Filter paper
Funnel
Graduated cylinder, 100-mL
Saturated $Ca(OH)_2$
0.1 M $Al_2(SO_4)_3$
Test tube
$AgNO_3$,
Ring stands
Burner
Round bottom flask
Ring
Wire gauze
Clamps
Thermometer
Stopper
Condenser
Erlenmeyer flask

**PROCEDURE**

1. A mixture of sand, dirt, salt, and water awaits you. Record observations from each of the following steps in your lab notebook. In a 250-mL beaker obtain approximately 100 mL of this mixture. Allow it to sit undisturbed for several minutes. Carefully pour the liquid portion into another beaker without disturbing the insoluble sediment. Discard the sediment into a lined trashcan. Keep the decanted solution for use in the next portion of the experiment.

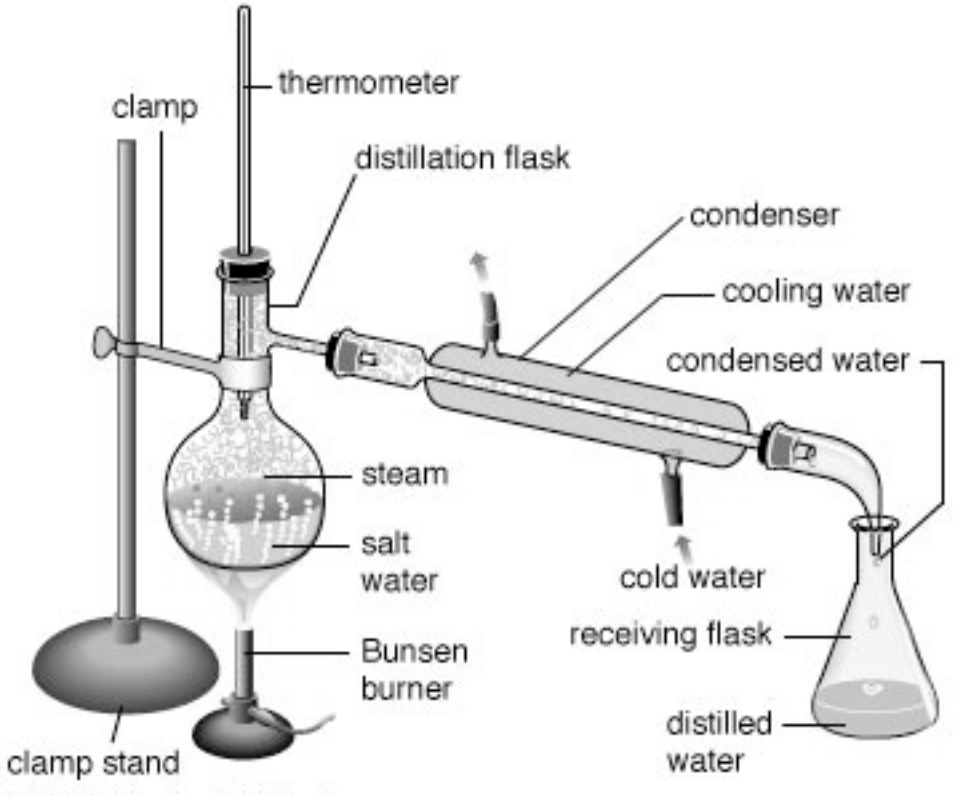
2. Flute a sheet of filter paper and place it in a funnel. Pour the solution obtained in step 1 through the filter paper, being careful not to allow it to overflow. Collect the solution in a 100-mL graduated cylinder.

3. From the reagents provided, mix 8 mL of saturated $Ca(OH)_2$ with 2 mL of 0.1 M $Al_2(SO_4)_3$ in a test tube and shake. Add this solution to the solution from step 2 and allow settling. The reaction occurring is:
   $Al_2(SO_4)_3 + Ca(OH)_2 \rightarrow$ _____?

4. Decant approximately 50 mL of clear solution from step 3 and add several drops of $AgNO_3$, sufficient to form a white precipitate indicating a positive test for dissolved chloride ions. The net ionic reaction occurring is:
   $Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$
   The chloride ions are present because the original solution contained some NaCl.

5. Place the solution from step 4 in the distillation flask and assemble your distillation apparatus (See illustration on following page). Distill the solution until approximately 10 mL of distillate have been collected. Remove the burner and test the distillate for the presence of dissolved $Cl^-$. Determine if your distillate was indeed purified.

**LABORATORY REPORT (25)**
Include your objective and *paraphrased* procedure. Your observations of each step should be included in full. There are no calculations or graphs associated with this experiment. Remember to write your conclusions.




*Distillation apparatus*

# LAW OF DEFINITE PROPORTIONS

Duration: 1 day

**INTRODUCTION**

Dalton's celebrated atomic theory included the concepts that all atoms of a given substance have the same weight and that combinations of atoms form compounds. The experimental information referred to as the Law of Definite Proportions supports these two concepts. Without exception, careful study of binary compounds bonded by conventional covalent or ionic bonding has shown that each compound always contains the same weight percentages of the two elements entering into chemical combinations to form the compound. The purpose of this experiment is to repeatedly synthesize a binary compound and to determine its empirical formula.

You will chemically combine copper and iodine. Copper is a soft metal that will be used in the form of a fine wire. Iodine is a solid that sublimes readily; that is, with gentle heating, the solid is converted to a vapor. In this experiment, the iodine vapor will be brought into contact with the copper wire. A new compound is formed in a chemical reaction, and it appears in the form of a white solid that adheres to the surface of the copper wire. The copper wire will then weigh more than it did originally because of the adherent compound. Note that the additional weight is due only to the iodide ions in the compound since the copper ions in the compound were originally part of the weight of the copper wire. The copper atoms have simply changed from an association with other copper atoms in the metal to an ionic association with iodide ions in the compound.

The chemical properties of ions of copper as they are found in the compound are different from the chemical properties of those copper atoms found in the metal. In particular, the ions of copper as they are found in the compound will react with a solution of sodium thiosulfate, $Na_2S_2O_3$, to form a new water-soluble substance containing the copper ions (copper wire will not react with the $Na_2S_2O_3$ solution). As a result, the copper compound dissolves while the unreacted copper atoms do not. Now, the copper wire, stripped of its coating of the compound, will weigh less than the original copper wire because some copper atoms have been removed from the surface. In fact, the difference in weight is just the weight of copper atoms that entered into chemical combination with iodide ions to form the white solid compound. Thus, the weight the copper wire gains upon being coated with the compound is due to the iodide ions in the compound; and the weight the copper wire has lost when the compound is stripped is due to the copper ions in the compound.

According to the Law of Definite Proportions, the weights of copper and iodine that combine to form the compound must always have the identical ratio.

**OBJECTIVES**

In this experiment, you will

- Determine the moles of copper and iodide ions present in a compound formed.
- Determine the formula of that copper iodide compound.

**MATERIALS**

#22 Cu wire
$I_2$ vapors
2 M $HNO_3$
Acetone
Wash bottle w/$dH_2O$
0.5 M $Na_2S_2O_3$
Balance

**PROCEDURE**

1. Wind a piece of #22 Cu wire tightly around a pen or pencil so as to form a spiral of wire. Leave about 5 cm of straight wire to be used as a handle. Form a short bend in the end of the handle so that the coil can be suspended from the rim of a test tube. Make certain that the wire is approximately 3 cm from the bottom of your test tubes and that the coils are not so compressed that they touch each other.

2. Dip the Cu wire in a dilute solution of $HNO_3$ (2 M) for about 15 seconds. Then rinse the coil with distilled water from the wash bottle. Repeat this process once. Dip the coil in acetone and let the coil air-dry.

3. Weigh the Cu coil on the top-loading balance by placing it gently on a piece of weighing paper that is already on the zeroed balance. Record all masses to the nearest 0.001 g in your data table.

4. You will find test tubes containing iodine being warmed in water baths so that iodine vapors rise near the top of the test tubes. Insert the coil in the test tube labeled with your lab station number and hook the handle of the coil over the rim of the test tube. Leave the coil in contact with the iodine vapors for 5-10 minutes. You should be able to see the copper iodide compound forming on the coil.

5. **Carefully** remove the wire from the test tube, taking care not to shake or jar it so severely that some of the coating is lost. Allow the wire to cool for 1 minute. Weigh the coated wire on the top-loading balance and record.

6. Immerse the coated coil in 0.5 M solution of $Na_2S_2O_3$ for 2-3 minutes. After the bright surface of the Cu reappears, remove the coil from the solution and rinse it in distilled water using the wash bottle.

7. Dip the coil in acetone, air-dry, weigh the coil on the analytical balance, and record.

8. Repeat the procedure at least two times.

**DATA TABLE**

| | Trial 1 | Trial 2 | Trial 3 |
|---|---|---|---|
| Mass of Cu wire before reacting (g) | | | |
| Mass Cu wire w/iodide ions after reacting (g) | | | |
| Mass of Cu wire after cpd is removed (g) | | | |

**LABORATORY REPORT (35)**

Include your objective and paraphrased procedure in paragraph format. Your observations of each step and all of your data should be clear. You need only show one calculation of each type in your lab report. Show all work performed in determining your average mole ratio of copper to iodine in the compound. Reduce this ratio to its simplest form, and you will have the empirical formula of your compound. In your conclusion address your findings and explain the following: If you had not removed the entire compound from the wire before beginning a new trial, how would your subsequent calculations have been affected?

# THE DETERMINATION OF THE PERCENT WATER IN A COMPOUND

Duration: 1 day

**INTRODUCTION**

The polarity of the water molecule, which makes it a great solvent for ionic compounds, causes water molecules to cling to the structure of solid substances. When this occurs, the trapped water molecules are called water of hydration and they become an integral part of the crystal structure.

There are many compounds that have a tendency to absorb water vapor from the air. These compounds are said to be *hygroscopic*, and can be used as moisture-reducing agents. Other compounds absorb such large quantities of water vapor that they will actually dissolve in their own water of hydration, a property known as *deliquescence*.

In this experiment, you will test a hygroscopic ionic compound to determine its water of hydration. Although the water molecules are securely attached to the ionic solid that you will test, they are susceptible to removal by heat. You will gently heat a sample of the compound to drive off the water of hydration. By measuring the mass of the sample before and after heating, you can determine the amount of water in the sample and calculate its water of hydration.

**OBJECTIVES**

In this experiment, you will

- Carefully heat a measured sample of a hygroscopic ionic compound.
- Determine the water of hydration of the compound.
- Complete the chemical formula of the compound.

**MATERIALS**

Crucible with cover
Crucible tongs
Spatula
Ring stand, ring, and clay triangle
Burner
Desiccator

One of the following compounds:
Magnesium sulfate, $MgSO_4 \bullet nH_2O$
Copper (II) sulfate, $CuSO_4 \bullet nH_2O$
Manganese (II) sulfate, $MnSO_4 \bullet nH_2O$
Sodium carbonate, $Na_2CO_3 \bullet nH_2O$
Balance

**PROCEDURE**

1. Measure and record the mass of a clean, dry crucible with cover. Obtain about 1–1.5 g of the selected compound and place it in the crucible. Use a spatula to break up any large pieces of the substance by pressing the pieces against the wall of the crucible. Measure and record the mass of the crucible, cover, and compound.

2. Set up a ring stand, ring, and clay triangle for heating the sample. Rest the crucible on the clay triangle. Tip the cover slightly so that it does not fit snugly on top of the crucible. Set up a lab burner and ignite the burner away from the crucible. Adjust the burner to get a small flame.

3. Gently heat the crucible for about ten minutes. Depending on the compound that you selected, the color of the sample may change significantly as the water of hydration is driven out of the crystals.

4. Turn off the burner. Cover the crucible and allow the sample to cool for about ten minutes.

5. When the crucible is cool enough to handle safely, measure and record the mass of the crucible, cover, and contents.

6. Heat the crucible of your sample for five more minutes, allow it to cool, and measure and record its mass.

7. Continue heating the sample for five minutes at a time, until you have two mass measurements that are within about 0.050 g of each other. If time constraints force you to complete the experiment on a second day, place the crucible in a desiccator until you can continue your work.

8. Dispose of your sample as directed.

**DATA TABLE**

| | |
|---|---|
| Compound selected for analysis | |
| Mass of crucible and cover (g) | |
| Mass of crucible, cover, and hydrated sample (g) | |
| Mass of crucible, cover, and dehydrated sample – 1st weighing (g) | |
| Mass of crucible, cover, and dehydrated sample – 2nd weighing (g) | |
| Mass of crucible, cover, and dehydrated sample – 3rd weighing (g) | |
| Mass of crucible, cover, and dehydrated sample – 4th weighing (g) | |

**LABORATORY REPORT (25)**

Include your objective and paraphrased procedure in paragraph format. All of your data collected should be included in your report. Show one calculation of each type (include such simple calculations as subtraction!). Your calculations should reveal the following: mass of hydrate, mass of dehydrated sample, mass of water evolved, moles of water in hydrate. Give the results of **all** calculations. Show your work in determining the formula of the hydrate. In your conclusion address your findings and explain the following: If you had not heated the sample long enough to remove all of the water of hydration, how would your subsequent calculations have been affected?

# DETERMINING THE MOLE RATIOS IN A CHEMICAL REACTION

Duration: 1 day

**INTRODUCTION**

A balanced chemical reaction equation gives the mole ratios of the reactants and the products as coefficients. When some of the chemical formulas are not known, an experiment must be conducted to help determine the mole ratios.

This experiment uses two common substances as the reactants: hypochlorite ion ($OCl^-$) from household bleach and thiosulfate ion ($S_2O_3^{2-}$), the active ingredient in a photographic "fixer" solution used to develop film. In the reaction, hypochlorite ions oxidize the thiosulfate ions according to the *unbalanced* and *incomplete* reaction equation below.

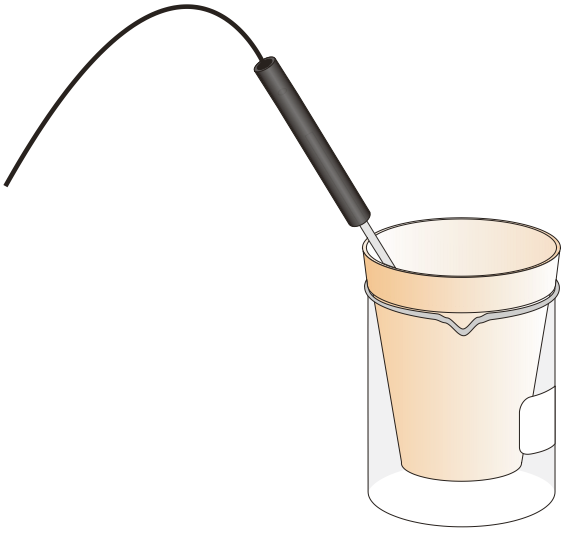
$$A\ OCl^- + B\ S_2O_3^{2-} \rightarrow \text{products}$$

It is possible to identify the coefficients, *A* and *B*, for the reactants, without knowing the products of the reaction. The process that you will use to determine the coefficients is called *continuous variations*. You will prepare a series of mixtures of the two reactants. Each mixture will have the same total volume . The reaction is exothermic. In procedure A, the mixture that generates the most heat energy will be the reaction that completely consumes both the hypochlorite and the thiosulfate ions. In procedure B, the point at which the temperature no longer changes will be the reaction that completely consumes both the hypochlorite and the thiosulfate ions. By choosing one of the procedures, you will use this mixture to establish the coefficients, and therefore the mole ratio, for the reaction.

**OBJECTIVES**

In this experiment, you will

- Measure the enthalpy change of a series of reactions.
- Determine the stoichiometry of an oxidation-reduction reaction in which the reactants are known but the products are unknown.



*Reaction vessel and temperature probe*

**MATERIALS**

Vernier LabQuest
Temperature Probe
Stands/buret clamps, burets
Beakers
0.50 M sodium hypochlorite, NaOCl, solution
0.50 M sodium thiosulfate, $Na_2S_2O_3$, solution in 0.2 M sodium hydroxide, NaOH
Styrofoam® cups

**PROCEDURE A**

1. Connect a Temperature Probe to Channel 1 of the Vernier computer interface.
2. Measure precisely 25.0 mL of the 0.50 M NaOCl solution. Pour this solution into a Styrofoam cup and nest the cup in a beaker to help stabilize the cup (see illustration on previous page).
3. Immerse the tip of the Temperature Probe in the Styrofoam cup of NaOCl solution.
4. Measure precisely 25.0 mL of the 0.50 M $Na_2S_2O_3$ solution. **Note:** Do not mix the two solutions yet.
5. Click ▸ Collect to begin data collection. Let the program gather and graph a few initial temperature readings, and then add the $Na_2S_2O_3$ solution. Gently stir the reaction mixture with the Temperature Probe.
6. Collect data for 3 minutes. Only if the temperature readings are no longer changing may you click ■ Stop or ▸ Collect to end data collection before 3 minutes have passed,.
7. Examine the graph to calculate and record the minimum and maximum temperatures for this volume ratio of the 0.50 M NaOCl solution and the 0.50 M $Na_2S_2O_3$ solution.
   - To determine these temperatures, click Analyze→Statistics→Temperature. The minimum and maximum temperatures are listed in the statistics box on the graph.
8. Rinse and dispose of the reaction mixture down the sink.
9. Open a new file by clicking File→New. Select Discard when prompted unless you wish to save the previous run on a flash drive.
10. Repeat the necessary steps to continue testing various ratios of the two solutions, keeping the total volume constant at 50.0 mL, until you have three measurements on either side of the ratio that produced the greatest temperature change. Record your volumes of each solution and your minimum and maximum temperature for each ratio.

**PROCEDURE B**

1. Connect a Temperature Probe to Channel 1 of the Vernier computer interface.
2. Measure precisely 5.0 mL of the 0.50 M $Na_2S_2O_3$ solution. Pour this solution into a Styrofoam cup and nest the cup in a beaker to help stabilize the cup (see illustration on previous page).
3. Immerse the tip of the Temperature Probe in the Styrofoam cup of $Na_2S_2O_3$ solution.
4. Measure precisely 44.0 mL of distilled water and add to the cup.
5. Measure precisely 1.0 mL of the 0.50 M NaOCl solution. **Note:** Do not mix the two solutions yet.

6. Click ▸ Collect to begin data collection. Let the program gather and graph a few initial temperature readings, and then add the NaOCl solution. Gently stir the reaction mixture with the Temperature Probe.

7. Collect data for 3 minutes. Only if the temperature readings are no longer changing may you click ■ Stop or ▸ Collect to end data collection before 3 minutes have passed,.

8. Examine the graph to calculate and record the minimum and maximum temperatures for this volume ratio of the 0.50 M NaOCl solution and the 0.50 M $Na_2S_2O_3$ solution.
   - To determine these temperatures, click Analyze→Statistics→Temperature. The minimum and maximum temperatures are listed in the statistics box on the graph.
9. Rinse and dispose of the reaction mixture down the sink.

10. Open a new file by clicking File→New. Select Discard when prompted unless you wish to save the previous run on a flash drive.

11. Repeat the necessary steps to continue testing various ratios of the two solutions, keeping $Na_2S_2O_3$ solution volume constant at 5.0 mL and the total volume constant at 50.0 mL,. Adjust the distilled water and NaOCl solution volumes until you have three constant temperatures. Record your volumes of each solution and your minimum and maximum temperature for each ratio.

**DATA TABLE**

| Volume $OCl^-$ (mL) | Volume $S_2O_3^{2-}$ (mL) | Minimum/Maximum Temperature (°C) |
|---|---|---|
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |
| | | |

**LABORATORY REPORT (40)**

Include your objective and paraphrased procedure in paragraph format. All of your data collected should be included in your report. Show one calculation of each type (include such simple calculations as subtraction!). Your calculations should reveal the following: temperature changes and mole ratios of reactants. Give the results of **all** calculations. Make a graph plotting volume of $OCl^-$ (mL) versus temperature change (°C). In your conclusion address your findings and the following:

- The molarities of the reactant solutions were equal in this experiment. Is this necessary, or even important, for the success of the experiment?
- Which solution was the limiting reactant in each trial?
- If the actual reaction between $OCl^-$ and $S_2O_3^{2-}$ in this basic medium produces sulfate ion, chloride ion, and water, what is the actual mole ratio of the $OCl^-$ and $S_2O_3^{2-}$? (*Hint…balance using the half-reaction method!)*
- Does the mole ratio that you determined in your experiment match the actual reaction equation's coefficients for the two reactants? Explain, especially if your mole ratios do not match the coefficients.

# OXIDATION-REDUCTION TITRATION WITH POTASSIUM DICHROMATE

Duration: 2 days

**INTRODUCTION**

The experiment illustrates how an oxidation-reduction reaction may be used to perform a volumetric analysis of iron. A solution of $K_2Cr_2O_7$ serves as the oxidizing agent to bring about the oxidation of an unknown amount of $Fe^{2+}$ to $Fe^{3+}$. An organic dye, which changes color at the redox potential of the equivalence point (when all of the ferrous ions have been oxidized), is used as an indicator.

The potassium dichromate has advantages over other oxidizing agents such as potassium permanganate in that its solutions are quite stable, even at higher temperatures, and that it is capable of oxidizing other species, such as the chloride ion, which are often present. The net ionic reaction is as follows:

$$Cr_2O_7^{2-} + 6\ Fe^{2+} + 14\ H^{1+} \rightarrow 2\ Cr^{3+} + 6\ Fe^{3+} + 7\ H_2O$$ ***How many e⁻ are exchanged?***

The redox indicator is a soluble organic compound that exists in two states of oxidation. These two forms are different colors. The barium salt of diphenylamine sulfonate serves as such an indicator, and the color change is from colorless to deep purple. This color change can be seen even in the presence of the green $Cr^{3+}$ ions that are formed by the reduction of the dichromate.

The sulfuric/phosphoric acid mixture is needed because it forms complex ions with the ferric ions, thereby lowering the $Fe^{2+}/Fe^{3+}$ potential. This gives greater differentiation between the $Fe^{2+}/Fe^{3+}$ potential and that of the two forms of the indicator. Thus, there is less chance of the indicator itself being oxidized before the oxidation of the $Fe^{2+}$ is complete.

**OBJECTIVES**

In this experiment, you will

- Perform an oxidation-reduction titration.
- Determine volume of $Cr_2O_7^{2-}$ required to reach the endpoint.
- Perform stoichiometry to determine the mass of $Fe^{2+}$ present in the sample and the percentage of $Fe^{2+}$ in the sample.

**MATERIALS**

Unknown containing $Fe^{2+}$
$K_2Cr_2O_7$
250-mL volumetric flask
Redox indicator
Erlenmeyer flasks
Concentrated sulfuric/phosphoric acid mixture
Stand/buret clamp, buret
Parafilm
Balance

**PROCEDURE**

**Part I Making $K_2Cr_2O_7$, weighing samples, and initial titration**

1. Weigh approximately 1.2 g of $K_2Cr_2O_7$ on the top-loading balance and record its exact mass to 0.001 g in your data table. Transfer the weighed amount of $K_2Cr_2O_7$ to a clean 250-mL volumetric flask and dissolve it in tap water, diluting the solution exactly.

2. Weigh three samples of approximately 1.0-1.4 g (recorded to the nearest 0.001 g in your data table) of your unknown. Place each into its own 250-mL Erlenmeyer flask.

3. To your lowest weight sample, add 75 mL of tap water, 8 drops of redox indicator, and 15 mL of the **concentrated** sulfuric/phosphoric acid mixture. ***Be extremely careful with this mixture.***

4. Transfer several mL of the $K_2Cr_2O_7$ to a clean buret and rinse it with several small portions of the solution. This ensures that the standard solution will not be diluted when the buret is filled. Fill the buret and zero it using a waste beaker.

5. Using a white piece of paper or a paper towel as a background, titrate the lowest weight flask to a deep purple endpoint. The solution will pass through a green color on the way to the endpoint. Be certain to obtain a definite purple color. Record the volume (to the nearest 0.01 mL) of $K_2Cr_2O_7$ needed to reach the endpoint in your data table.

6. Cover your remaining two samples with Parafilm until Day 2.

**Part II Titrating two remaining samples.**

1. Add 75 mL of tap water, 8 drops of redox indicator, and 15 mL of the **concentrated** sulfuric/phosphoric acid mixture to the lower weight sample.

2. Place several mL of the $K_2Cr_2O_7$ in the clean buret and rinse it with several small portions of the solution. Fill the buret and zero it using a waste beaker.

3. Titrate this flask to the purple endpoint and record the volume (to the nearest 0.01 mL) of $K_2Cr_2O_7$ needed in your data table.

4. Repeat these three steps with the remaining sample.

5. Empty all containers of their contents and rinse at least three times with water. Allow buret to dry upside down and open on its stand. All other glassware should be placed on the drying racks or on paper towels.

**DATA TABLE**

| | Trial 1 | Trial 2 | Trial 3 |
|---|---|---|---|
| Mass of $K_2Cr_2O_7$ (g) | | | |
| Mass of unknown (g) | | | |
| Volume of $K_2Cr_2O_7$ (mL) | | | |

**LABORATORY REPORT (35)**

Include your objective and paraphrased procedure in paragraph format. All of your data collected should be included in your report. Show one calculation of each type. Your calculations should reveal the following: molarity of $K_2Cr_2O_7$, mass of $Fe^{2+}$ present in each unknown sample, percentage of $Fe^{2+}$ in each unknown sample, the calculation of the average percentage of iron in the sample. In your conclusion, address your findings and any potential errors.

# EXPLORING GAS LAWS

Duration: 2 days

**INTRODUCTION**

The purpose of this investigation is to conduct a series of experiments, each of which illustrates a different gas law. Four properties of gases will be investigated: pressure, volume, temperature, and number of moles. By assembling the equipment, conducting the appropriate tests, and analyzing your data and observations, you will be able to describe the gas laws, both qualitatively and mathematically.

**OBJECTIVES**

In this experiment, you will

- Conduct a set of experiments, each of which illustrates a gas law.
- Gather data to identify the gas law described by each activity.
- Complete the calculations necessary to evaluate the gas law in each activity.
- From your results, derive a single mathematical relationship that relates pressure, volume, temperature, and number of moles.

**MATERIALS**

Vernier LabQuest
Large-volume container for water bath
Vernier Gas Pressure Sensor
125-mL Erlenmeyer flask
Temperature Probe
Hot plate
20-mL gas syringe
Ice
Plastic tubing with two Luer-lock connectors
100-mL graduated cylinder
Rubber stopper assembly with two-way valve

**PRE-LAB EXERCISE**

Review each of the four parts of this experiment before starting your work. You will need to decide the best way to conduct the testing, so it is wise to make some plans before you begin. You may wish to conduct a test run without collecting data, in order to observe how the experiment will proceed.

In each part of the experiment, you will investigate the relationship between two of the four possible variables, the other two being constant. In this pre-lab exercise, sketch a graph that describes your hypothesis as to the mathematical relationship between the two variables; e.g., direct relationship or inverse relationship.

**Part I** Pressure, P, and volume, V (temperature and number of moles constant).

**Part II** Pressure, P, and absolute temperature, T (volume and number of moles constant).

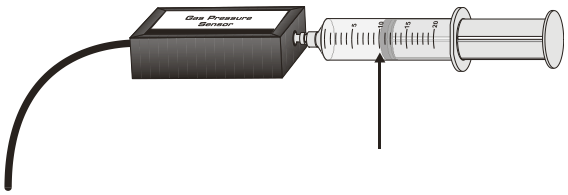
**Part III** Volume, V, and absolute temperature, T (pressure and number of moles constant).

**Part IV** Pressure, P, and number of moles, n (volume and absolute temperature constant).

**PROCEDURE**

**Part I Pressure and Volume**

1. Position the piston of a plastic 20 mL syringe so that there will be a measured volume of air trapped in the barrel of the syringe. Attach the syringe to the valve of the Gas Pressure Sensor as shown below. A gentle half turn should connect the syringe to the sensor securely. **Note:** Read the volume at the front edge of the inside black ring on the piston of the syringe, as indicated by the arrow.

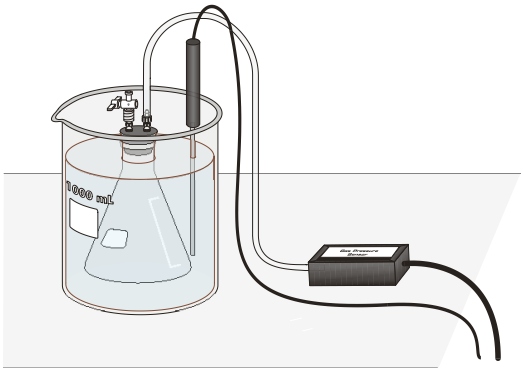



2. Connect the Gas Pressure Sensor to Channel 1 of the LabQuest. Change your pressure units to atm.

3. Measure and record the pressure of the air in the syringe at various volumes. The best results are achieved by collecting at least six data points.

**Part II Pressure and Absolute Temperature**

In this experiment, you will study the relationship between the absolute temperature of a gas sample and the pressure it exerts. Using the apparatus shown below, you will place an Erlenmeyer flask containing an air sample in a water bath and you will vary the temperature of the water bath.




1. Connect the Gas Pressure Sensor to Channel 1 and a Temperature Probe to Channel 2 of the LabQuest.

2. Assemble the apparatus shown above. Be sure that all fittings are airtight. Make sure the rubber stopper and flask neck are dry, then twist and push hard on the rubber stopper to ensure a tight fit.

3. Collect and record pressure data (atm) at several different temperatures (K). Set up water baths in the large- volume container, as you need to do so.

**Part III Volume and Absolute Temperature**

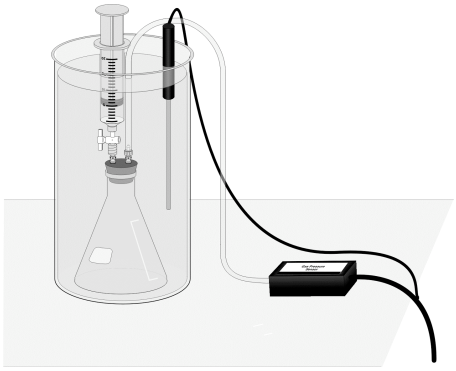
In this experiment, you will study the relationship between the volume of a gas sample and its absolute temperature. Using the apparatus shown on the next page, you will place an Erlenmeyer flask containing an air sample in a water bath and you will vary the temperature of the water bath. Keep some of these factors in mind as you plan your procedure.

• If you are starting with a cold-water bath, set the piston at the 0 mL mark on the syringe. This will allow the gas volume to be increased in warmer water baths.

• The temperature of the water bath cannot be increased by more than 30-40 degrees from your starting temperature.

• Even though you are not plotting pressure, it is important to monitor pressure in the Meter to ensure that it remains constant.

• It is important to know the *total* volume of air in the flask *and* the syringe. The volume of the flask, up to the bottom of rubber stopper, can be accurately measured using a graduated cylinder. For the estimated volume of the tubing (from the rubber stopper to the Gas Pressure Sensor box), as well as in the valve below the bottom of the syringe, use a value of ~4 mL.



1. Ensure that the Gas Pressure Sensor is plugged into to Channel 1 and the Temperature Probe is plugged into Channel 2 of the LabQuest.

2. Assemble the apparatus shown above. Be sure that all fittings are air-tight. Make sure that the rubber stopper and flask neck are dry, then twist and push hard on the rubber stopper to ensure a tight fit. Be sure that the water level is at least as high as the confined air in the syringe.

3. Collect and record volume data (mL) at several different temperatures (K). Set up water baths in the large-volume container as you need them, ranging from ice water to hot water. **Part IV**

**Part IV. Pressure and Number of Moles**

In this experiment, you will study the relationship between the number of moles in a gas sample and the pressure exerted.

• You can use the same setup as in the previous trial, although the water bath and Temperature Probe are optional. (Temperature must be constant, so choose a convenient temperature to run the experiment.)

• You might be wondering how you are going to count moles for this section. Here is a hint. Avogadro's hypothesis states that, "Equal volumes of gases, at the same temperature and pressure, contain equal numbers of moles." Therefore, *if* you keep the temperature and volume constant during the experiment, you can assume that gas volumes are proportional to numbers of moles. Instead of entering a total number of moles, you

could enter a total *volume* of gas that has been compressed into the flask. For example, 120 mL worth of moles could be entered as 120 moles, 140 mL worth of moles would be entered as 140 moles.)

1. Ensure that the Gas Pressure Sensor is plugged into Channel 1 of the LabQuest.

2. Collect and pressure data with several different numbers of moles introduced into the system.

**DATA ANALYSIS**

1. For each of the four parts of the experiment, construct a graph representing the data that you collected. Remember to place the independent variable on the x-axis!

2. For each of the four parts of the experiment, write an equation using the two variables and a proportionality constant, $k$ (e.g., for Part I, $P = k \times V$ if direct, or $P = k/V$ if inverse).

3. Calculate the constant, $k$, for each of the four gas laws that you tested. This value can be an average for each of the data pairs in each part of the experiment.

4. Based on the mathematical relationship and equation that you obtained in Step 1 above for each part of the experiment, combine all four variables into a final equation. This "combined equation" will contain $P$, $T$, $V$, and $n$, as well as a new proportionality constant, $K$. Be sure to explain how you obtained your result (how you combined the equations).

**LABORATORY REPORT (60)**

Include your objective and paraphrased procedure in paragraph format. All of your data collected should be included in your report. Show one calculation of each type. Your calculations should reveal the following: proportionality constant for each part (k), proportionality constant for combined equation ($K$). Your graphs should be included with your lab report. In your conclusion address your findings and compare them to your predictions made in the pre-lab exercise.

# MOLECULAR WEIGHT OF A VOLATILE LIQUID

Duration: 1 day

**INTRODUCTION**

The most common instrument of the determination of molecular weights in modern chemical research is the **mass spectrometer**. Such an instrument permits very precise determination of molecular weight and also gives a great deal of structural information about the molecule being analyzed; this is of great help in the identification of new or unknown compounds.

Mass spectrometers, however, are extremely expensive and take a great deal of time and effort to calibrate and maintain. In other words, you don't have one, nor will you get one in the near future! For these reasons, many of the classical methods of molecular weight determination are still widely applied. In this experiment, a common modification of the ideal gas law will be used in the determination of the molecular weight of a liquid that is easily evaporated.

The ideal gas law indicates that the observed properties of a gas sample ($P$, $V$, and $T$) are directly related to the quantity of the gas in the sample ($n$). For a given container of fixed volume at a particular temperature and pressure, only one possible quantity of gas can be present in the container: $n = PV/RT$. By careful measurement of the weight of the gas sample under study in the container, the molecular weight of the gas sample can be calculated. The molecular weight, $M$, merely represents the number of grams of the volatile substance per mole: $M = \text{g}/n$

In this experiment, a small amount of volatile liquid will be placed in a flask of known volume. The flask will be heated in a boiling water bath and its contents will be equilibrated with atmospheric pressure. From the volume in the flask used, the temperature of the boiling water bath, and the atmospheric pressure, the number of moles of gas contained in the flask may be calculated. From the mass of the liquid required to fill the flask with vapor when it is in the boiling water bath, the molecular weight of the liquid may be calculated.

A major assumption is made in this experiment that may affect your results. You assume that the vapor of the liquid behaves as an ideal gas. Actually, a vapor behaves least like an ideal gas under conditions near which the vapor would liquefy. The unknown liquid provided in this experiment has been chosen because its vapor most readily approaches ideal gas behavior.

**OBJECTIVES**

In this experiment, you will

- Evaporate a sample of a liquid substance and measure certain physical properties of the substance as it condenses
- Determine the molar mass of the liquid substance

**MATERIALS**

Vernier LabQuest
Large-volume container for water bath
Temperature Probe
Ring stand
Utility clamps
Al foil
Unknown volatile liquid
250-mL Erlenmeyer flask
Burner
Top-loading balance
Triple-beam balance

**PROCEDURE**

1. Prepare a water bath apparatus using a 600-mL beaker filled to half-capacity. Test the volume of water in the beaker by gently placing a 250-mL Erlenmeyer flask in the beaker and making certain the flask can be immersed to the 200-mL mark. Add or remove water from the beaker as needed. Heat the water to boiling. Once it boils, adjust the flame as needed to prevent splashing.

2. Clean and completely dry the inside of the flask. The inside of the flask must be *completely dry*, since any water present will vaporize under the conditions of the experiment and will adversely affect the results. Wrap a square of Al foil over the mouth of the flask and weigh the flask and its cover to the nearest 0.001 g.

3. Obtain 3-4 mL, exact volume is not needed, of the unknown liquid from the fume hood. Place all of the unknown liquid in the flask and replace the cover. Make certain that the foil cover is tightly crimped around the rim of the flask and that it does not extend down the neck of the flask. At this point, notify me that you are ready for your hole to be punched in the foil cover.

4. Immerse the flask assembly in the boiling water so that the 200-mL mark on the flask is immersed. Clamp the neck of the flask to the ring stand using a test tube clamp. Plug the temperature probe into Channel 1 of the Lab Quest and immerse the probe in the water bath.

5. Watch the unknown liquid *very carefully*. The liquid will begin to evaporate rapidly, and its volume will decrease. The amount of liquid placed in the flask is much more than will be necessary to fill the flask with vapor at the boiling water temperature. Excess vapor will be observed escaping through the pinhole in the cover.

6. When it appears that all of the unknown liquid has vaporized, and the flask is filled with vapor, continue to heat for 1-2 more minutes. Then *very carefully* using the clamp on the neck of the flask to protect your hands, remove the flask from the boiling water.

7. Place the flask on the lab top, remove the clamp (without disturbing the foil cover), and allow the flask to cool to room temperature. Liquid will reappear in the flask as the vapor in the flask cools. While the flask is cooling, measure and record the exact temperature (to the nearest 0.1°C) of the boiling water in the beaker and record the barometric pressure in the laboratory.

8. When the flask has cooled to room temperature (after about 5 minutes), carefully dry the outside of the flask to remove any droplets of water. Weigh the flask, foil cover, and condensed vapor to the nearest 0.001 g.

9. Repeat steps 3-8. The weight of the flask after the second sample of unknown liquid is vaporized and condensed should agree with the first determination within 0.050 g. If it does not agree, do a third trial.

10. When two acceptable determinations of vapor weight needed to fill the flask have been obtained, remove the foil cover from the flask and rinse the flask.

11. Fill the flask to the very rim with tap water, cover with the foil, and weigh the flask ***on the triple beam balance*** to the nearest 0.1 g. Determine the temperature of the tap water in the flask. Using the density of water at the temperature of the water in the flask (obtained from the *Handbook of Chemistry and Physics*, p. F-5) and the weight of water the flask contains, you will determine the exact volume of the flask.

**DATA ANALYSIS**

1. Using the volume of the flask, the temperature of the boiling water bath, and the barometric pressure, calculate the number of moles of vapor the flask is capable of containing. *Think about the appropriate units needed for the volume, temperature, and pressure.*

2. Using the mass of the unknown vapor contained in the flask (use an average if your weights were not identical but were within 0.05 g), and the number of moles of vapor contained in the flask, calculate the molecular weight of the unknown liquid.

**LABORATORY REPORT (40)**

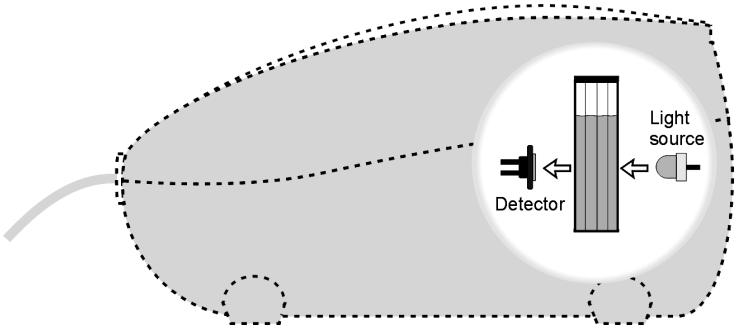
Include your objective and *paraphrased* procedure. All data should be easily read. Show one calculation of each type. Address both steps of Data Analysis. Remember to write your conclusions. You will be graded in part on your accuracy of the molecular weight of the liquid. I will give you the actual molecular weight when I pass your report back to you. At that time, you should add a calculation of your percentage error in your lab notebook.

# DETERMINING THE CONCENTRATION OF A SOLUTION: BEER'S LAW

Duration: 1 day

**INTRODUCTION**

The primary objective of this experiment is to determine the concentration of an unknown copper (II) sulfate solution. You will use a Colorimeter (a side view is shown in Figure 1) to measure the concentration of each solution. In this experiment, red light from the LED light source will pass through the solution and strike a photocell. A higher concentration of the colored solution absorbs more light (and transmits less) than a solution of lower concentration. The Colorimeter monitors the light received by the photocell as percent transmittance.




*Colorimeter*

You will prepare five copper (II) sulfate solutions of known concentration (standard solutions). Each solution is transferred to a small, rectangular cuvette that is placed into the Colorimeter. The amount of light that penetrates the solution and strikes the photocell is used to compute the absorbance of each solution. When you graph absorbance *vs*. concentration for the standard solutions, a direct relationship should result. The direct relationship between absorbance and concentration for a solution is known as *Beer's law*.

You will determine the concentration of an unknown $CuSO_4$ solution by measuring its absorbance with the Colorimeter. By locating the absorbance of the unknown on the vertical axis of the graph, the corresponding concentration can be found on the horizontal axis. The concentration of the unknown can also be found using the slope of the Beer's law curve.

**OBJECTIVES**

In this experiment, you will

- Prepare and test the absorbance of five standard copper (II) sulfate solutions.
- Calculate a standard curve from the test results of the standard solutions.
- Test the absorbance of a copper (II) sulfate solution of unknown molar concentration.
- Calculate the molar concentration of the unknown $CuSO_4$ solution.

**MATERIALS**

Vernier LabQuest
Vernier Colorimeter
Graduated cylinders
Test tubes
Cuvette
Beakers
0.40 M copper (II) sulfate, $CuSO_4$, solution
Copper (II) sulfate, $CuSO_4$, unknown solution
Stands/buret clamps, burets
Distilled water
Test tube rack
Stirring rod
Tissues (lint-free)

**PROCEDURE**

1. Obtain small volumes of 0.40 M $CuSO_4$ solution and distilled water in separate beakers.

2. Label five clean, dry, test tubes 1–5. Use burets to prepare five standard solutions according to the chart below. Thoroughly mix each solution with a stirring rod. Clean and dry the stirring rod between uses.

| Trial number | 0.40 M $CuSO_4$ (mL) | Distilled $H_2O$ (mL) | Concentration (M) |
|---|---|---|---|
| 1 | 2.0 | 8.0 | 0.080 |
| 2 | 4.0 | 6.0 | 0.16 |
| 3 | 6.0 | 4.0 | 0.24 |
| 4 | 8.0 | 2.0 | 0.32 |
| 5 | 10.0 | 0 | 0.40 |

3. Connect the Colorimeter to LabQuest in Channel 1 and choose New from the File menu.

4. Calibrate the Colorimeter. Prepare a *blank* by filling an empty cuvette 3/4 full with distilled water. Place the blank in the cuvette slot of the Colorimeter and close the lid. Press the < or > buttons on the Colorimeter to set the wavelength to 635 nm (Red). Then calibrate by pressing the CAL button on the Colorimeter. When the LED stops flashing, the calibration is complete.

5. On the Meter screen, tap Mode. Change the data-collection mode to Events with Entry. Enter the Name (Concentration) and Units (mol/L). Select OK.

6. You are now ready to collect absorbance-concentration data for the five standard solutions. Start data collection. Remove the cuvette from your Colorimeter and pour out the water. Using the solution in test tube 1, rinse the cuvette twice with ~1 mL amounts, and then fill it 3/4 full. Wipe the outside with a tissue, place it in the Colorimeter, and close the lid.
   - When the absorbance readings have stabilized, tap Keep and enter **0.080** as the concentration. Select OK. The absorbance and concentration values have now been saved for the first solution. Discard the cuvette contents as directed.
   - Using the solution in test tube 2, rinse the cuvette twice with ~1 mL amounts, and then fill it 3/4 full. Wipe the outside, place it in the Colorimeter, and close the lid. When the absorbance readings have stabilized, tap Keep and enter **0.16** as the concentration in mol/L. Select OK.
   - Repeat Part these steps for test tube 3 (0.24 M), test tube 4 (0.32M), and the stock 0.40 M $CuSO_4$ in test tube 5

- **Note:** Do not test the unknown solution until Step 8. Stop data collection to view a graph of absorbance *vs.* concentration. To examine the data pairs on the displayed graph, select any data point. As you tap each point, the absorbance and concentration values of each data point are displayed to the right of the graph. Record the absorbance values in your data table.

7. Display a graph of absorbance *vs.* concentration with a linear regression curve. Choose Curve Fit from the Analyze menu. Select Linear as the Fit Equation. The linear-regression statistics for these two data columns are displayed for the equation in the form $y = mx + b$ where $x$ is concentration, $y$ is absorbance, $a$ is the slope, and $b$ is the y-intercept.
   - **Note:** One indicator of the quality of your data is the size of $b$. It is a very small value if the regression line passes through or near the origin. The correlation coefficient, $r$, indicates how closely the data points match up with (or *fit*) the regression line. A value of 1.00 indicates a nearly perfect fit.
   - Select OK. The graph should indicate a direct relationship between absorbance and concentration, a relationship known as Beer's law. The regression line should closely fit the five data points *and* pass through (or near) the origin of the graph. Record the best-fit line equation for the standard solutions in your data table.

8. Determine the absorbance value of the unknown $CuSO_4$ solution. Obtain about 5 mL of the *unknown* $CuSO_4$ in another clean, dry, test tube. Record the number of the unknown in your data table. Rinse the cuvette twice with the unknown solution and fill it about 3/4 full. Wipe the outside of the cuvette, place it into the Colorimeter, and close the lid. Tap Meter and monitor the absorbance value displayed on the screen. When this value has stabilized, record it in the data table. Dispose of any of the remaining solutions as directed.

**DATA TABLE**

| Trial | Concentration (mol/L) | Absorbance |
|---|---|---|
| 1 | 0.080 | |
| 2 | 0.16 | |
| 3 | 0.24 | |
| 4 | 0.32 | |
| 5 | 0.40 | |
| 6 | Unknown number ___ | |

Best-fit line equation: _____

**DATA ANALYSIS**

1. Make a graph showing the data and linear-regression equation for the standard solutions.

2. Determine the concentration of the unknown $CuSO_4$ solution. Explain how you made this determination.

3. Describe an alternate method for determining the molar concentration of your unknown sample of copper (II) sulfate solution, using the standard data.

**LABORATORY REPORT (40)**

Include your objective and *paraphrased* procedure. All data should be easily read. Address all three points of Data Analysis within your report. Show your calculation of the unknown concentration. Remember to write your conclusions. You will be graded in part on your accuracy of the concentration of the unknown. I will give you the actual concentration when I pass your report back to you. At that time, you should add a calculation of your percentage error in your lab notebook.

# DETERMINING THE ENTHALPY OF A CHEMICAL REACTION

Duration: 1 day

**INTRODUCTION**

All chemical reactions involve an exchange of heat energy; therefore, it is tempting to plan to follow a reaction by measuring the enthalpy change ($\Delta H$). However, it is often not possible to directly measure the heat energy change of the reactants and products (the system). We can measure the heat change that occurs in the surroundings by monitoring temperature changes. If we conduct a reaction between two substances in aqueous solution, then the enthalpy of the reaction can be indirectly calculated with the following equation.

$$q = C_p \times m \times \Delta T$$

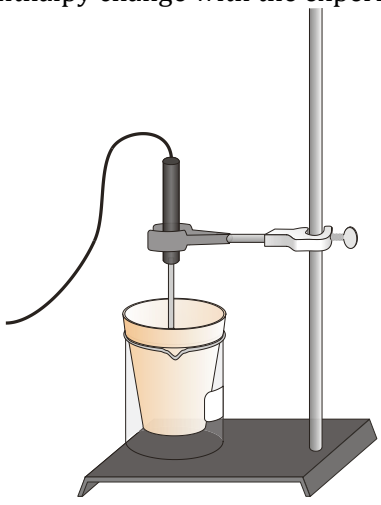
The term $q$ represents the heat energy that is gained or lost. $C_p$ is the specific heat of water, $m$ is the mass of water, and $\Delta T$ is the temperature change of the reaction mixture. The specific heat and mass of water are used because water will either gain or lose heat energy in a reaction that occurs in aqueous solution. Furthermore, according to a principle known as Hess's law, the enthalpy changes of a series of reactions can be combined to calculate the enthalpy change of a reaction that is the sum of the components of the series.

In this experiment, you will measure the temperature change of two reactions, and use Hess's law to determine the enthalpy change, $\Delta H$ of a third reaction. You will use a Styrofoam cup nested in a beaker as a calorimeter, as shown in the figure below. For purposes of this experiment, you may assume that the heat loss to the calorimeter and the surrounding air is negligible.

**OBJECTIVES**

In this experiment, you will

- Use Hess's law to determine the enthalpy change of the reaction between aqueous ammonia and aqueous hydrochloric acid.
- Compare your calculated enthalpy change with the experimental results.



*Calorimeter*

**MATERIALS**

| | |
|---|---|
| Vernier LabQuest | 2.0 M HCl |
| Temperature Probe | 2.0 M NaOH |
| Glass stirring rod | 2.0 M $NH_4Cl$ |
| Styrofoam cup | 2.0 M $NH_3$ |
| Beaker | Ring stand |
| Graduated cylinders | Clamp |

**PRE-LAB EXERCISE**

You will conduct the following three reactions in this experiment. In the space provided below, write the balanced net ionic reaction equations from the descriptions. Use the table of thermodynamic data in your text (or another approved resource) to calculate the molar enthalpy of the reactions.

Reaction 1: An aqueous solution of sodium hydroxide reacts with an aqueous solution of hydrochloric acid, yielding water.

Reaction 2: An aqueous solution of sodium hydroxide reacts with an aqueous solution of ammonium chloride, yielding aqueous ammonia, $NH_3$, and water.

Reaction 3: An aqueous solution of hydrochloric acid reacts with aqueous ammonia, $NH_3$, yielding aqueous ammonium chloride.

| Reaction | Balanced reaction equation | $\Delta H$ (kJ/mol) |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |

**PROCEDURE**

1. Connect the Temperature Probe to LabQuest and choose New from the File menu.

2. Use a utility clamp to suspend the Temperature Probe from a ring stand as shown.

**Part I Conduct the Reaction Between Solutions of NaOH and HCl**

3. Nest a Styrofoam cup in a beaker, as shown. Measure out 50.0 mL of 2.0 M HCl solution into the foam cup. Lower the Temperature Probe into the solution. **CAUTION:** *Handle the hydrochloric acid with care. It can cause painful burns if it comes in contact with the skin*.

4. Measure 50.0 mL of NaOH solution, but do not add it to the HCl solution yet. **CAUTION:** *Handle the sodium hydroxide solution with care.*

5. Conduct the reaction.
   - Start data collection and obtain the initial temperature of the HCl solution.
   - After three or four readings have been recorded at the same temperature, add the 50.0 mL of NaOH solution to the Styrofoam cup all at once. Use a glass stirring rod to stir the reaction mixture continuously until the temperature reaches a peak and begins to fall.
   - Data collection will end after three minutes. If the temperature readings are no longer changing, you may stop the trial early.
   - To examine the data pairs on the displayed graph, select any data point. As you move the examine line, the temperature values of each data point are displayed to the right of the graph.
   - Record the initial and maximum temperatures in your data table.
6. Rinse and dry the Temperature Probe, Styrofoam cup, and the stirring rod. Dispose of the solution as directed.

**Part II Conduct the Reaction Between Solutions of NaOH and $NH_4Cl$**

7. Measure 50.0 mL of 2.0 M NaOH solution into a nested Styrofoam cup. Lower the tip of the Temperature Probe into the cup of NaOH solution.
8. Measure 50.0 mL of 2.0 M $NH_4Cl$ solution, but do not add it to the NaOH solution yet.
9. Conduct the reaction.
   - Repeat Step 5 with the 50.0 mL of $NH_4Cl$ solution
   - Examine the graph as before to determine and record the initial and maximum temperatures of the reaction.
10. Rinse and dry the Temperature Probe, Styrofoam cup, and the stirring rod. Dispose of the solution as directed.

**Part III Conduct the Reaction Between Solutions of HCl and $NH_3$**

11. Measure 50.0 mL of 2.0 M HCl solution into a nested Styrofoam cup. Lower the tip of the Temperature Probe into the cup of HCl solution.
12. Measure 50.0 mL of 2.0 M $NH_3$ solution, but do not add it to the HCl solution yet.
13. Conduct the reaction.
    - Repeat Step 5 with the 50.0 mL of $NH_3$ solution
    - Examine the graph as before to determine and record the initial and maximum temperatures of the reaction.

**DATA TABLE**

| | Reaction 1 | Reaction 2 | Reaction 3 |
|---|---|---|---|
| Maximum temperature (°C) | | | |
| Initial temperature (°C) | | | |
| Temperature change ($\Delta T$) | | | |

**DATA ANALYSIS**

1. Calculate the amount of heat energy, $q$, produced in each reaction. Use 1.03 g/mL for the density of all solutions. Use the specific heat of water, 4.18 J/(g•°C), for all solutions.

2. Calculate the enthalpy change, $\Delta H$, for each reaction in terms of kJ/mol of each reactant.

3. Use your answers from #2 above and Hess's law to determine the experimental molar enthalpy for Reaction 3.

4. Use Hess's law, and the accepted values of $\Delta H$ in the Pre-Lab Exercise to calculate the $\Delta H$ for Reaction 3. How does the accepted value compare to your experimental value?

5. Does this experimental process support Hess's law? Suggest ways of improving your results.

**LABORATORY REPORT (35)**

Include your objective and *paraphrased* procedure. All data should be easily read. Address all five points of Data Analysis within your report. Show one calculation of each type and the results of each calculation. Remember to write your conclusions.

# VAPOR PRESSURE AND THE HEAT OF VAPORIZATION

Duration: 2 days

**INTRODUCTION**

When a liquid is placed in a container, and the container is sealed tightly, a portion of the liquid will evaporate. The newly formed gas molecules exert pressure in the container, while some of the gas condenses back into the liquid state. If the temperature inside the container is held constant, then at some point equilibrium will be reached. At equilibrium, the rate of condensation is equal to the rate of evaporation. The pressure at equilibrium is called *vapor pressure*, and will remain constant as long as the temperature in the container does not change.

In mathematical terms, the relationship between the vapor pressure of a liquid and temperature is described in the Clausius-Clayperon equation,

$$\ln P = \frac{-\Delta H_{vap}}{R}\left(\frac{1}{T}\right) + C$$

where ln $P$ is the natural logarithm of the vapor pressure, $\Delta H_{vap}$ is the heat of vaporization, $R$ is the universal gas constant (8.31 J/mol•K), $T$ is the Kelvin temperature, and $C$ is a constant not related to heat capacity. Thus, the Clausius-Clayperon equation not only describes how vapor pressure is affected by temperature, but it relates these factors to the heat of vaporization of a liquid. $\Delta H_{vap}$ is the amount of energy required to cause the evaporation of one mole of liquid at constant pressure.

In this experiment, you will introduce a specific volume of a volatile liquid into a closed vessel, and measure the pressure in the vessel at several different temperatures. By analyzing your measurements, you will be able to calculate the $\Delta H_{vap}$ of the liquid.

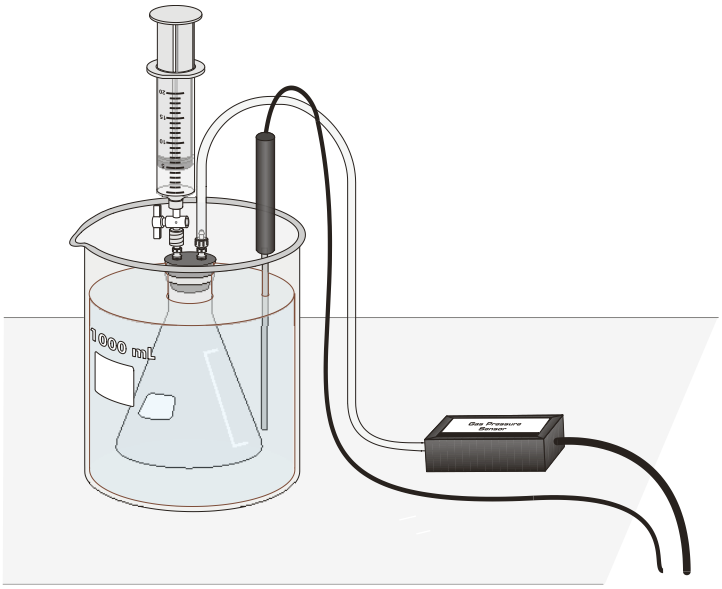
**OBJECTIVES**

In this experiment, you will

- Measure the pressure inside a sealed vessel containing a volatile liquid over a range of temperatures.
- Determine the relationship between pressure and temperature of the volatile liquid.
- Calculate the heat of vaporization of the liquid.

**MATERIALS**

| | |
|---|---|
| Vernier LabQuest | 20-mL syringe |
| Vernier Gas Pressure Sensor | two 125-mL Erlenmeyer flasks |
| Temperature Probe | ethanol, $CH_3CH_2OH$ |
| rubber stopper assembly | beakers |
| plastic tubing with two connectors | hot plate |




*Vapor Pressure Apparatus*

**PROCEDURE**

1. Use a hot plate to heat ~200 mL of water in a 400 mL beaker.

2. Prepare a room temperature water bath in a large beaker. The bath should be deep enough to completely cover the gas level in the 125 mL Erlenmeyer flask.

3. Connect the Gas Pressure Sensor and Temperature Probe to the LabQuest. Choose New from the File menu.

4. Use the clear tubing to connect the white rubber stopper to the Gas Pressure Sensor. (About one-half turn of the fittings will secure the tubing tightly.) Twist the white stopper snugly into the neck of the Erlenmeyer flask to avoid losing any of the gas that will be produced as the liquid evaporates. **Important**: Open the valve on the white stopper.

5. Change the data-collection mode to Selected Events.

6. Your first measurement will be of the pressure of the air in the flask and the room temperature. Place the Temperature Probe near the flask. When the pressure and temperature readings stabilize, record these values in the first column (Initial) of your data table.

7. Condition the Erlenmeyer flask and the sensors to the water bath. Place the Temperature Probe in the room temperature water bath. Place the Erlenmeyer flask in the water bath. Hold the flask down into the water bath to the bottom of the white stopper. After 30 seconds, close the valve on the white stopper.

8. Obtain a small amount of ethanol. Draw 3 mL of ethanol into the 20-mL syringe. Thread the syringe onto the valve on the white stopper.

9. Add ethanol to the flask. Open the valve below the syringe containing the 3 mL of ethanol. Push down on the plunger of the syringe to inject the ethanol. Quickly pull the plunger back to the 3 mL

mark. Close the valve below the syringe. Carefully remove the syringe from the stopper so that the stopper is not moved.

10. Gently rotate the flask in the water bath for a few seconds, using a motion similar to slowly stirring a cup of coffee or tea, to accelerate the evaporation of the ethanol.

11. Monitor and collect temperature and pressure data. Start data collection. Hold the flask steady once again. Monitor the pressure and temperature readings. When the readings stabilize, select Keep.

12. Add a small amount of hot water, from the beaker on the hot plate, to warm the water bath by 3–5°C. Stir the water bath slowly with the Temperature Probe. Monitor the pressure and temperature readings. When the readings stabilize, select Keep.

13. Repeat Step 12 until you have completed five total trials. Add enough hot water for each trial so that the temperature of the water bath increases by 3–5°C, but do not warm the water bath beyond 40°C because the pressure increase may pop the stopper out of the flask. If you must remove some of the water in the bath, do it carefully so as not to disturb the flask.

14. After you have recorded the fifth set of readings, open the valve to release the pressure in the flask. Remove the flask from the water bath and take the stopper off of the flask. Dispose of the ethanol as directed.

15. Stop data collection. Tap Table to see the temperature and pressure measurements. Record the pressure readings, as $P_{total}$, and the temperature readings in your data table.

**DATA TABLE**

| | Initial | Trial 1 | Trial 2 | Trial 3 | Trial 4 | Trial 5 |
|---|---|---|---|---|---|---|
| $P_{total}$ (kPa) | | | | | | |
| $P_{air}$ (kPa) | | | | | | |
| $P_{vap}$ (kPa) | | | | | | |
| Temperature (°C) | | | | | | |

**DATA ANALYSIS**

1. The $P_{air}$ for Trials 2–5 must be calculated because the temperatures were increased. As you warmed the flask, the air in the flask exerted pressure that you must calculate. Use the gas law relationship shown below to complete the calculations. Remember that all gas law calculations require Kelvin temperature. Use the $P_{air}$ from Trial 1 as $P_1$ and the Kelvin temperature of Trial 1 as $T_1$.

$$\frac{P_1}{T_1} = \frac{P_2}{T_2}$$

2. Calculate and record the $P_{vap}$ for each trial by subtracting $P_{air}$ from $P_{total}$.

3. Prepare a graph of $P_{vap}$ (y-axis) *vs*. Celsius temperature (x-axis). Does the plot follow the expected trend of the effect of temperature on vapor pressure? Explain.

4. Prepare a second graph. The vertical axis for this graph will be the natural logarithm of $P_{vap}$, ln $P_{vap}$, and the horizontal axis will be the reciprocal of Kelvin temperature ($1/T$). Calculate the linear regression (best-fit line) equation for this graph. Calculate $\Delta H_{vap}$ from the slope of the linear regression using the Clausius-Clayperon equation.

5. The accepted value of the $\Delta H_{vap}$ of ethanol is 42.32 kJ/mol. Compare your experimentally determined value of $\Delta H_{vap}$ with the accepted value.

**LABORATORY REPORT (45)**

Include your objective and *paraphrased* procedure. All data should be easily read. Address all five points of Data Analysis within your report. Show one calculation of each type and the results of all calculations. Remember to write your conclusions.

# USING FREEZING POINT DEPRESSION TO FIND MOLAR MASS

Duration: 2 days

**INTRODUCTION**

When a solute is dissolved in a solvent, the freezing temperature is lowered in proportion to the number of moles of solute added. This property, known as freezing-point depression, is a *colligative property*; that is, it depends on the ratio of solute and solvent particles, not on the nature of the substance itself. The equation that shows this relationship is

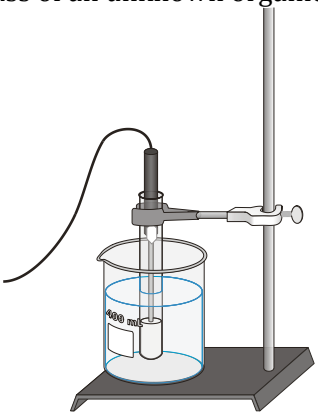
$$\Delta T = k_f m i$$

where $\Delta T$ is the freezing point depression (in °C) , $k_f$ is the freezing point depression constant for a particular solvent, (in kg°C/mol), m is the molality of the solution (in mol solute/kg solvent), and *i* is the van't Hoff factor (1 since you will use covalent substances).

**OBJECTIVES**

In this experiment, you will

- Determine the freezing temperature of the pure solvent, Butylated hydroxytoluene (BHT).
- Determine the freezing temperature of a mixture of BHT and *para*-dichlorobenzene.
- Calculate the freezing point depression of the mixture.
- Calculate the freezing point constant for BHT.
- Calculate the molar mass of an unknown organic solid.



*Freezing Point Depression Apparatus*

**MATERIALS**

Vernier LabQuest
Vernier Temperature Probe
Test tubes
Beaker
Ring stand/clamp
Butylated Hydroxytoluene, $C_{15}H_{24}O$
*Para*-dichlorobenzene, $C_6H_4Cl_2$
Unknown organic solid
Hot plate

**PROCEDURE**

1. Connect the Temperature Probe to LabQuest and choose New from the File menu.

2. On the Meter screen, tap Rate. Change the data-collection rate to 1 sample/second and the data-collection length to 600 seconds.

**Part I Determine the Freezing Temperature of Pure BHT**

3. Add about 300 mL of tap water to a beaker. Place the beaker on the base of the ring stand. Heat the beaker with the water using a hot plate on setting "5" or at "204°C."

4. Measure about 4 g of BHT and place into a test tube. Record the exact mass of BHT used to the nearest 0.001 g.

5. Clamp the test tube in the water bath and insert the temperature probe. Heat the water bath to about 90°C.

6. Start data collection. Remove the test tube from the water bath.

7. With a very slight up-and-down motion of the Temperature Probe, *continuously* stir the BHT for the ten-minute duration of the experiment.

8. When data collection is complete, use a hot water bath to melt the BHT enough to safely remove the Temperature Probe. Carefully wipe any excess BHT liquid from the probe with a paper towel.

9. The freezing temperature can be determined by finding the mean temperature in the portion of the graph with nearly constant temperature. Select the data point at the beginning of the flat portion of the graph and drag across the flat portion to select the region. Choose Statistics from the Analyze menu. Record the mean (average) temperature as the freezing temperature of pure BHT.

10. Record or store the data from the first run on a flash drive.

**Part II Determine the Freezing Point of a Solution of BHT and *Para*-dichlorobenzene**

11. Measure about 0.5 g of *para*-dichlorobenzene onto a piece of weighing paper and record its exact mass to 0.001g.

12. Place the *para*-dichlorobenzene into the test tube containing the BHT. Heat the mixture in the hot water bath until the substances are melted, stirring well to ensure a homogeneous mixture.

13. Repeat steps 1-2 and 6-10.

14. Place the test tube in a 600-mL beaker in the fume hood.

**Part III Determine the Freezing Point of a Solution of BHT and Unknown Organic Solid**

15. Measure about 4 g of fresh BHT into the test tube and record the exact mass of BHT used to the nearest 0.001 g.

16. Measure about 0.5 g of your unknown onto a piece of weighing paper and record its exact mass to the nearest 0.001 g..

17. Place the unknown into the test tube containing the BHT. Heat the mixture in the hot water bath until the substances are melted, stirring well to ensure a homogeneous mixture.

18. Repeat steps 1-2 and 6-10.

19. Obtain your test tube from the previous day from the fume hood. Heat both test tubes in your hot water bath until the mixtures are melted. Pour the melted substances out onto the provided crumpled newspaper in the fume hood.

20. Rinse both test tubes with acetone from the fume hood before using soap and water to clean them.

**DATA TABLE**

| | |
|---|---|
| Mass of BHT (g) | |
| Freezing temperature of pure BHT (°C) | |
| Mass of *para*-dichlorobenzene (g) | |
| Freezing point of BHT/ *para*-dichlorobenzene (°C) | |
| Freezing point of BHT/ unknown (°C) | |

**DATA ANALYSIS**

1. Graph your data plotting time of cooling versus temperature in °C, you should show the cooling curves for the pure BHT, the BHT/ *para*-dichlorobenzene solution, and the BHT/unknown solution on the same graph. Thus, you should have three distinct curves on your graph. Make them different colors or different line styles.

2. Determine the freezing points of the BHT, the BHT/ *para*-dichlorobenzene solution and the BHT/unknown solution.

3. Determine the values of $\Delta T_f$ for both the BHT and the solution from your data and your graphs.

4. Calculate the $k_f$ for BHT from the data of the BHT/ *para*-dichlorobenzene solution.

5. Using the calculated $k_f$ for BHT, calculate the molar mass of the unknown solute.

**LABORATORY REPORT (45)**

Include your objective and *paraphrased* procedure. All data should be easily read. Address all five points of Data Analysis within your report. Show one calculation of each type and the results of all calculations. Remember to write your conclusions. You will be graded in part on your accuracy. I will give you the actual molar mass of the unknown and its identity when I pass your report back to you. At that time, you should add a calculation of your percentage error in your lab notebook.

# KINETICS OF A REACTION

Duration: 3 days

**INTRODUCTION**

In this experiment you will study the kinetics of a chemical reaction. The reaction is called a "clock" reaction because of the means of observing the reaction rate. The reaction involves the oxidation of iodide ion by bromate ion in the presence of an acid:

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

The reaction is somewhat slow at room temperature. Its rate depends on the concentration of the reactants and on the temperature. The rate law is a mathematical expression that relates the reaction rate to the concentrations of reactants. If you express the rate of reaction as the rate of decrease in concentration of bromate ion, the rate law has the form:

$$\text{Rate} = \frac{-\Delta[BrO_3^-]}{\Delta t} = k[I^-]^x\,[BrO_3^-]^y\,[H^+]^z$$

where the brackets refer to the molar concentration of the indicated species. The rate is equal to the change in concentration of the bromate ion, $-\Delta[BrO_3^-]$ , divided by the change in time for the reaction to occur, $\Delta t$. The term *"k"* is the rate constant for the equation, and changes as temperature changes. The exponents *x, y,* and *z* are called the "orders" of the reaction with respect to the indicated substance and show how the concentration of each substance affects the rate of reaction. One purpose of the experiment is to determine the total rate law for the process. To do this, you must measure the rate, evaluate the rate constant, *k,* and determine the order of the reaction for each reactant, the values of *x, y,* and *z.* A second goal is to determine the activation energy for the reaction. Lastly you will see the effect a catalyst has on the reaction rate.

To find the rate of the reaction, you need some way of measuring the rate at which one of the reactants is used, or the rate at which one of the products is formed. The method that you will use is based on the rate at which iodine forms. If thiosulfate ions are added to the solution, they react with iodine as it forms in this way:

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

Rxn. 1 is somewhat slow. Rxn. 2 proceeds extremely rapidly, so that as quickly as iodine is produced in Rxn. 1, it is consumed in Rxn. 2. Rxn. 2 continues until all of the thiosulfate is used. After that, iodine begins to increase in concentration in solution. If some starch is present, iodine will react with the starch to form a deep blue-colored complex that is readily apparent. Carrying out Rxn. 1 in the presence of thiosulfate ion and starch produces a chemical "clock." When the thiosulfate is consumed, the solution turns blue.

In all of your reactions you will use the same quantity of thiosulfate ion. The blue color appears when all of the thiosulfate is used. An examination of Rxns. 1 and 2 shows that 6 moles of $S_2O_3^{2-}$ are needed to react with the $I_2$ formed from 1 mole of $BrO_3^-$. Knowing the amount of thiosulfate used allows the calculation of the amount of $I_2$ that is formed, and also the amount of $BrO_3^-$ that has reacted at the time of the color change. The reaction rate is expressed as the decrease in concentration of $BrO_3^-$ ion divided by the time it takes for the blue color to appear.

The experiment is designed so that the amounts of the reactants that are consumed are small in comparison with the total quantities present. This means that the concentration of reactants is almost unchanged during the reaction, and, therefore, the reaction rate is almost constant during this time.

The experiment is designed using a microscale procedure. Only 12 drops of reactants delivered from capillary droppers will be used for each measurement. The steps involved are as follows: Determine the volume of a drop of solution. This must be done so that the number of moles of thiosulfate ion can be found, and so the amount of bromate ions that react can be calculated. Find the order of the reaction for each of the reactants, and determine the rate constant. You will do this by carrying out an experiment at specific concentrations of each of the reactants and measuring the reaction rate. Then you will change the concentration of one reactant and observe how the reaction rate changes. This will be repeated for each reactant. This data allows the calculation of the order of each reactant. Once the orders are known, the value of the rate constant can be calculated.

Next you will study the relation between the rate of the reaction and temperature. Reaction rates always increase as temperature goes up. By measuring how the rate changes as the temperature is varied, you can determine the activation energy, $E_a$, for the reaction. The equation giving this relation is:

$$\ln k = \frac{-E_a}{RT} + \ln A$$

where ln $k$ is the natural logarithm of the rate constant, $E_a$ is the activation energy, $R$ is the gas constant, 8.314 J/K-mol, and $T$ is the temperature on the Kelvin scale. $A$ is a constant called the frequency factor which you will not need to determine. This equation follows the straight-line relationship: $y = mx + b$. A plot of $T^{1-}$ versus the natural logarithm of $k$ will give a straight-line graph. The slope of the graph will be $-E_a/R$, and you will use the slope to determine the activation energy.

In the last part of the experiment you will observe the effect of a catalyst on the rate of the reaction.

**OBJECTIVES**

In this experiment, you will

- Conduct the reaction of iodide, bromate, and hydrogen ions using various concentrations.
- Determine the order of each reactant.
- Determine the rate law for the reaction.
- Calculate the activation energy for the reaction.
- Observe the effects of a catalyst on the reaction.

**MATERIALS**

| | |
|---|---|
| Vernier LabQuest | 0.010 M KI |
| Vernier Temperature Probe | Distilled $H_2O$ |
| micropipets | 0.10 M HCl |
| 96-well plate | 2% Starch |
| Beakers | 0.0010M $Na_2S_2O_3$ |
| Hot-plate | 0.040 M $KBrO_3$ |
| ice | of 0.10M $Cu(NO_3)_2$ |

**PROCEDURE**

**Part I Find the volume of a drop of solution.**

1. Find the mass of a small beaker.

2. While holding the dropper vertically, deliver 5 drops of water into the beaker and find the total mass. Add an additional 5 drops of water and again determine the total mass. Deliver 5 more drops and again find the total mass.

3. Assume that the density of each of the dilute solutions that will be used is the same as that of water, 1g/mL.

4. Calculate the volume of one drop of water.

**Part II Determine the reaction rate and calculate the rate law.**

The table that follows shows the reagent quantities to be used in carrying out the reactions needed. Because you do not want the reaction to start until you are ready, *be sure that the $KBrO_3$ solution is the last solution added.* It is important to use care in measuring the solutions. Since the total solution volume is quite small, even one extra drop can cause a substantial change in concentrations.

| Exp. # | KI 0.010M | $H_2O$ | HCl 0.10M | Starch 2% soln | $Na_2S_2O_3$ 0.0010M | $KBrO_3$ 0.040M |
|---|---|---|---|---|---|---|
| 1 | 2 drops | 4 drops | 2 drops | 1 drop | 1 drop | 2 drops |
| 2 | 4 drops | 2 drops | 2 drops | 1 drop | 1 drop | 2 drops |
| 3 | 6 drops | 0 drops | 2 drops | 1 drop | 1 drop | 2 drops |
| 4 | 2 drops | 2 drops | 2 drops | 1 drop | 1 drop | 4 drops |
| 5 | 2 drops | 0 drops | 2 drops | 1 drop | 1 drop | 6 drops |
| 6 | 2 drops | 2 drops | 4 drops | 1 drop | 1 drop | 2 drops |
| 7 | 2 drops | 0 drops | 6 drops | 1 drop | 1 drop | 2 drops |
| 8 | 3 drops | 1 drop | 3 drops | 1 drop | 1 drop | 3 drops |

*Reagent Quantities*

It is necessary to use consistently good technique to obtain reproducible data. Hold droppers vertically and be sure no air bubbles are introduced. Since such small quantities of reagents are used, it is very easy to repeat measurements. Practice your technique by carrying out the first experiment at least three times (more, if necessary) until your values are reproducible. *Calculation of the orders of reactants are all based on the values obtained for the first experiment, so be sure to get reproducible data from the beginning.* All other experiments should be carried out at least twice.

A study of the Reagent Quantities table shows that all experiments contain the same total number of drops of solution. Only one drop of sodium thiosulfate, $Na_2S_2O_3$, and one drop of starch solution are added to each well. In experiments 1, 2, and 3, the concentration of potassium iodide, KI, is gradually increased while all other volumes remain constant. Experiments 1, 4, and 5 have an increasing concentration of potassium bromate, $KBrO_3$. Experiments 1, 6, and 7 show an increase in the concentration of hydrochloric acid, HCl. Experiment 8 will be a test to see if calculated orders of reactants agree with experimental values.

5. Measure the drops of solutions required for experiment 1 in one of the wells on top of a piece of white paper. Be sure to add $KBrO_3$ last. Stir the mixture thoroughly with a toothpick. This is very important, because it is impossible to achieve good mixing in the small well without stirring. Begin timing the reaction as soon as the $KBrO_3$ is added.

6. Record the time required for the first blue color to appear.

7. Repeat the trials until consistently reproducible values are obtained.

8. Record the room temperature as the temperature of these reactions.

9. Empty the well plate, rinse with water and shake to dry the wells. Use detergent and a cotton swab, if necessary, to be sure the wells are clean and dry for each experiment.

10. Carry out the experiments with solution volumes described in Experiments 2 through 8.

**Part III Determine the activation energy.**
In this part of the lab investigation, the reaction will be carried out at several temperatures using the concentration of Exp. 1. The temperatures will be about 40°C, 20°C, 10°C, and 0°C. Use your value for Exp. 1 at room temperature for the "about 20°C"—*when graphing, you will use the actual temperatures*—measurement.

11. Prepare a shallow water bath of about 40°C in a beaker.

12. Repeat the procedure using the concentrations in Exp. 1. Mix all of the solutions except the $KBrO_3$ and place the well strip in the warm water bath. Place the pipet containing the $KBrO_3$ in the bath as well.

13. After about 5 minutes in the bath, add the 2 drops of $KBrO_3$ to the well, stir, and time the reaction until the blue color first appears. Leave the strip in the bath while you are timing.

14. Repeat the quantities from Exp. 1 for the other two water baths at approximately 10°C, and 0°C.

15. Record the time and temperature for each.

**Part IV Observe the effect of a catalyst on the rate.**
16. Repeat the procedure for Exp. 1, but this time add 1 drop of 0.10M $Cu(NO_3)_2$ and only 3 drops of water to the mixture. The total volume will still be 12 drops.

17. Record the reaction time.

*Clean all strips and glassware used.*

**DATA ANALYSIS**

**Find the volume of a drop of solution.**

1. Find the average mass of 1 drop of water after each addition of 5 drops of water. Remember to subtract the mass of the beaker!

3. Determine the average of the three average masses of 1 drop of water. This will be your volume of 1 drop to be used in further calculations.

**Determine the reaction rate and calculate the rate law.**

Place a table similar to the one below in your lab notebook to facilitate your calculations:

| | ----------Time (s)---------- | | | | | | | Initial Concentrations (M) | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Exp.# | Trial 1 | Trial 2 | Trial 3 | Trial 4 | Trial 5 | Temp (°C) | Rxn Rate (M/s) | $[I^-]$ | $[BrO_3^-]$ | $[H^+]$ |
| 1 | | | | | | | | | | |
| 2 | | | | | | | | | | |
| 3 | | | | | | | | | | |
| 4 | | | | | | | | | | |
| 5 | | | | | | | | | | |
| 6 | | | | | | | | | | |
| 7 | | | | | | | | | | |
| 8 | | | | | | | | | | |

*Reaction rate data and preliminary calculations*

3. Calculate the rate

Recall that the rate will be expressed as $\frac{-\Delta[BrO_3^-]}{\Delta t}$

In each reaction, there is 1 drop of 0.0010M $Na_2S_2O_3$ solution. Calculate the number of moles of $Na_2S_2O_3$ present in 1 drop by:

*volume of 1 drop (in L) x 0.0010mol* $Na_2S_2O_3$/L = moles $S_2O_3^{2-}$ions

The blue color begins to appear when all of the thiosulfate ion is consumed. Examination of Exp. 1 and 2 allows you to calculate the moles of $BrO_3^-$ which react as all of the $S_2O_3^{2-}$ ion is used:

$$mol\ S_2O_3^{2-} \times \frac{1\ mol\ I_2}{2\ mol\ S_2O_3^{2-}} \times \frac{1 mol\ BrO_3^-}{3\ mol\ I_2} = mol\ BrO_3^-\ reacted$$

The value of $-\Delta[BrO_3^-]$ in all reactions, since all experiments have a total volume of 12 drops is:

$$-\Delta[BrO_3^-] = \frac{mol\ BrO_3^-\ reacted}{volume\ of\ 12\ drops}$$

The rate of each reaction can be found by dividing $-\Delta[BrO_3^-]$ by the average number of seconds required for the reaction to take place.

4. Calculate the initial concentrations

Calculate the initial concentration of each reactant for each experiment. This will not be the same as the concentration of the starting solution because combining the reactants dilutes all of the solutions. On dilution, the number of moles of reactant stays the same, therefore:

$$\#\ moles = V_{concentrated} \times M_{concentrated} = V_{dilute} \times M_{dilute}$$

where $V_{concentrated}$ and $M_{concentrated}$ are the volume and molarity of the starting, concentrated solutions; and $V_{dilute}$ and $M_{dilute}$ are the volume and molarity of the diluted reaction mixtures. (Wow, eh?!) Since volumes will be proportional to the number of drops of solution used, you can substitute drops for volume.

For example, in Exp. 1, the initial $[I^-]$ is found in this way:

$$[I^-]_o = \frac{2\ drops \times 0.010M\ KI}{12\ drops\ solution} = 0.0017M$$

Find the initial concentration of each reactant and place it in your table.

5. Calculate the order of each reactant

Next, you need to find the values for the exponents *x, y,* and *z* from your rate law expression. This investigation is designed so that the concentration of one ion changes while the others remain constant. Comparing values in Exp. 1, 2, and 3, you see that Exp. 2 has 2 times the $[I^-]$ as Exp. 1, and Exp. 3 has 3 times the $[I^-]$ as Exp. 1.

Substitute the values for Exp. 1 and 2 into the equation: rate = $k[I^-]^x [BrO_3^-]^y [H^+]^z$. Divide the first equation by the second equation. Notice that most of the terms will cancel out and you will have:

$$\frac{Rate_1}{Rate_2} = \frac{[I^-]_1^x}{[I^-]_2^x}$$

Divide and solve for *x*. Round the value of *x* to the nearest integer. Repeat the calculations using Exp. 1 and 3 to confirm your value for *x*.

Next, use the same manner with Exp. 1, 4, and 5 to find the value for *y*. Lastly, use Exp. 1, 6, and 7 to find the value of *z*.

6. Calculate the rate constant

Substitute data from each experiment into the rate law equation to find the value of *k*. Report the average value of *k*. Do not forget to include the units of *k*.

Exp. 8 is a check on your data. Substitute the concentrations of the reactants for Exp. 8 into the rate law that you determined and calculate the value of the rate of reaction. How does this calculated rate compare with the measured rate for Exp. 8 from your table?

7. Write the rate law for the reaction.

**Determine the activation energy.**
Place a table similar to the one below in your lab notebook to facilitate your calculations:

| | | | ------Time of rxn (s)------ | | | | | |
|---|---|---|---|---|---|---|---|---|
| Temp (°C) | Temp (K) | Temp$^{-1}$ (1/K) | Trial 1 | Trial 2 | Avg, | Rxn. Rate (M/s) | Rate constant, *k* | ln *k* |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |

*Activation energy data and preliminary calculations*

8. Construct a graph of $T^{1-}$ (1/K) versus the natural logarithm of *k*.

9. Calculate the activation energy using the slope of the best-fit line

**LABORATORY REPORT (90)**
Include your objective and *paraphrased* procedure. All data should be easily read…you might reproduce the tables that also include some of your calculated values for ease of reading. Address all nine steps of the Data Analysis. Show one calculation of each type. Report the results of the remaining calculations. Remember to write your conclusions. Do state the effect of your catalyst on the rate. I will give you the actual rate law when I pass your report back to you. There will be no percent error calculation for this experiment.

# DETERMINING AN EQUILIBRIUM CONSTANT

Duration: 2 days

**INTRODUCTION**

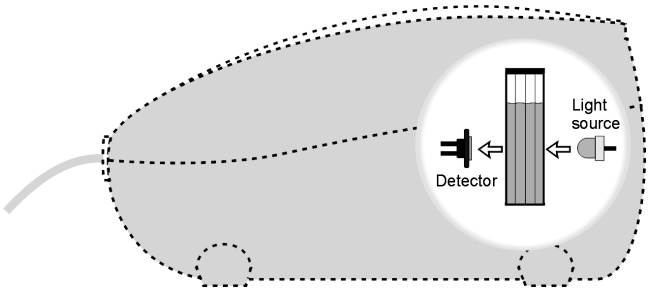
Chemical reactions occur to reach a state of equilibrium. The equilibrium state can be characterized by quantitatively defining its equilibrium constant, $K_{eq}$. In this experiment, you will determine the value of $K_{eq}$ for the reaction between iron (III) ions and thiocyanate ions, $SCN^-$.

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

The equilibrium constant, $K_{eq}$, is defined by the equation shown below.

$$K_{eq} = \frac{[FeSCN^{2+}]}{[Fe^{3+}][SCN^-]}$$

To find the value of $K_{eq}$, which depends only upon temperature, it is necessary to determine the molar concentration of each of the three species in solution at equilibrium. You will use a colorimeter to help you measure the concentrations (see Figure 1). The amount of light absorbed by a colored solution is proportional to its concentration. The red $FeSCN^{2+}$ solution absorbs blue light, and it will be analyzed at 470 nm (blue light).




*Colorimeter*

In order to successfully evaluate this equilibrium system, it is necessary to conduct three separate tests. First, you will prepare a series of standard solutions of $FeSCN^{2+}$ from solutions of varying concentrations of $SCN^-$ and constant concentrations of $H^+$ and $Fe^{3+}$ that are in stoichiometric excess. The excess of $H^+$ ions will ensure that $Fe^{3+}$ engages in no side reactions (to form $FeOH^{2+}$, for example). The excess of $Fe^{3+}$ ions will make the $SCN^-$ ions the limiting reagent, thus all of the $SCN^-$ used will form $FeSCN^{2+}$ ions. The $FeSCN^{2+}$ complex forms slowly, taking at least one minute for the color to develop. It is best to take absorbance readings after a specific amount of time has elapsed, between two and four minutes after preparing the equilibrium mixture. Do not wait much longer than four minutes to take readings, however, because the mixture is light sensitive and the $FeSCN^{2+}$ ions will slowly decompose.

In Part II of the experiment, you will analyze a solution of unknown $[SCN^-]$ by using the same procedure that you followed in Part I. In this manner, you will determine the molar concentration of the $SCN^-$ solution.

Third, you will prepare a new series of solutions that have varied concentrations of the $Fe^{3+}$ ions and the $SCN^-$ ions, with a constant concentration of $H^+$ ions. You will use the results of this test to accurately evaluate the equilibrium concentrations of each species.

**OBJECTIVES**
In this experiment, you will

- Prepare and test standard solutions of $FeSCN^{2+}$ in equilibrium.
- Test solutions of $SCN^-$ of unknown molar concentration.
- Determine the molar concentrations of the ions present in an equilibrium system.
- Determine the value of the equilibrium constant, $K_{eq}$, for the reaction.

**MATERIALS**

| | |
|---|---|
| Vernier LabQuest | 0.200 M iron (III) nitrate, $Fe(NO_3)_3$, solution in 1.0 M $HNO_3$ |
| Vernier Colorimeter | |
| Temperature Probe (optional) | 0.0020 M iron (III) nitrate, $Fe(NO_3)_3$, solution in 1.0 M $HNO_3$ |
| Plastic cuvette | |
| Test tubes | 0.0020 M thiocyanate, $SCN^-$ |
| Ring stands/clamps/burets | Test tube rack |
| Beakers | Potassium thiocyanate, KSCN solution of unknown concentration |
| Plastic Beral pipets | |
| Distilled water | Tissue |

**PRE-LAB EXERCISE**
For the solutions that you will prepare in Step 1 of Part I below, calculate the $[FeSCN^{2+}]$. Presume that all of the $SCN^-$ ions react. In Part I of the experiment, mol of $SCN^-$ = mol of $FeSCN^{2+}$. Thus, the calculation of $[FeSCN^{2+}]$ is: mol $FeSCN^{2+}$ ÷ L of *total* solution. Record these values in the table below.

| Beaker number | $[FeSCN^{2+}]$ |
|---|---|
| 1 | 0.00 M |
| 2 | |
| 3 | |
| 4 | |
| 5 | |

**PROCEDURE**

**Part I Prepare and Test Standard Solutions**

1. Connect the Colorimeter to LabQuest and choose New from the File menu. Calibrate the Colorimeter.
   - Prepare a *blank* by filling an empty cuvette 3/4 full with distilled water.
   - Place the blank in the cuvette slot of the Colorimeter and close the lid.
   - Press the < or > buttons on the Colorimeter to set the wavelength to 470 nm (Blue). Then calibrate by pressing the CAL button on the Colorimeter. When the LED stops flashing, the calibration is complete.

2. On the Meter screen, tap Mode. Change the data-collection mode to Events with Entry. Enter the Name (Concentration) and Units (mol/L). Select OK.

3. Label five beakers 1–5. **CAUTION:** *Fe(NO3)3 solutions in this experiment are prepared in 1.0 M HNO3 and should be handled with care.* Prepare five solutions according to the chart below. Do not make more than one solution at a time. Use a buret to distribute the necessary

amounts of each substance. Mix each solution thoroughly. Measure and record the temperature of one of the above solutions to use as the temperature for the equilibrium constant, $K_{eq}$.

| Beaker number | 0.200 M $Fe(NO_3)_3$ (mL) | 0.0020 M $SCN^-$ (mL) | $H_2O$ (mL) |
|---|---|---|---|
| 1 | 5.0 | 0.0 | 45.0 |
| 2 | 5.0 | 2.0 | 43.0 |
| 3 | 5.0 | 3.0 | 42.0 |
| 4 | 5.0 | 4.0 | 41.0 |
| 5 | 5.0 | 5.0 | 40.0 |

4. You are now ready to collect absorbance data for the standard solutions. **Note**: Take readings within 4 minutes of preparing the mixtures.
   a. Start data collection.

   b. Empty the water from the cuvette. Using the solution in Beaker 1, rinse the cuvette twice with ~1 mL amounts and then fill it 3/4 full. Wipe the outside with a tissue, place it in the colorimeter, and close the lid. When the absorbance readings stabilize, tap Keep and enter the concentration of $FeSCN^{2+}$ (from your Pre-Lab exercise) for the first trial. Select OK to continue.

   c. Discard the cuvette contents as directed. Rinse and fill the cuvette with the solution in Beaker 2. Wipe the outside with a tissue, place it in the colorimeter, and close the lid. Follow the procedure in Part b of this step to measure the absorbance and enter the concentration of this solution.

   d. Repeat this process to find the absorbance of the solutions in Beakers 3, 4, and 5.

   e. Stop data collection to view a graph of absorbance *vs.* concentration. To examine the data pairs on the displayed graph, select any data point. As you tap each point, the absorbance and concentration values of each data point are displayed to the right of the graph.

5. Record the absorbance values, for each of the five solutions, in your data table.

6. Display a graph of absorbance vs. concentration with a linear regression curve.
   a. Choose Curve Fit from the Analyze menu.

   b. Select Linear as the Fit Equation. The linear-regression statistics are displayed to the right of the graph for the equation in the form $y = mx + b$.

   c. Record the best-fit line equation in your data table and select OK.

**Part II Test an Unknown Solution of $SCN^-$**

7. Obtain 5.0 mL of the unknown into a clean and dry beaker. Add precisely 5.0 mL of 0.200 M Fe(NO3)3 and 40.0 mL of distilled water to the beaker. Stir the mixture thoroughly.

8. Using the solution in the beaker, rinse a cuvette twice with ~1 mL amounts and then fill it ¾ full. Wipe the outside with a tissue, place it in the colorimeter, and close the lid.

9. Determine the concentration of the unknown.
   a. Tap Meter. Monitor the absorbance readings on the screen.

   b. When the readings stabilize, record the absorbance value for your unknown in your data table.

   c. Remove and clean the cuvette.

   d. Tap Graph and choose Interpolate from the Analyze menu.

   e. Trace the linear regression equation to find the concentration of your unknown at the absorbance displayed on the meter.

**Part III Prepare and Test Equilibrium Systems**

10. Prepare five test tubes of solutions according to the chart below. Follow the necessary steps from Part I to test the absorbance values of each mixture. Record the results in your data table. **Note:** You are using 0.0020 M $Fe(NO_3)_3$ in this test.

| Test tube number | 0.0020 M $Fe(NO_3)_3$ (mL) | 0.0020 M $SCN^-$ (mL) | $H_2O$ (mL) |
|---|---|---|---|
| 1 | 3.00 | 0.00 | 7.00 |
| 2 | 3.00 | 2.00 | 5.00 |
| 3 | 3.00 | 3.00 | 4.00 |
| 4 | 3.00 | 4.00 | 3.00 |
| 5 | 3.00 | 5.00 | 2.00 |

11. To get good data for the calculation of $K_{eq}$, you must determine the net absorbance of the solutions in test tubes 2–5. To do this, subtract the absorbance reading for test tube 1 from the absorbance readings of test tubes 2–5, and record these values as net absorbance in your data table.

**DATA TABLE**

**Parts I and II**

| Beaker number | Absorbance |
|---|---|
| 1 | |
| 2 | |
| 3 | |
| 4 | |
| 5 | |
| Unknown, Part II | |

Best-fit line equation for the Part I standard solutions: _____

**Part III**

| Test tube number | Absorbance | Net absorbance |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |

**DATA ANALYSIS**

1. (Part II) Use the best-fit line and the absorbance reading for your unknown solution to determine the [$SCN^-$].

2. (Part II) Compare your experimental [$SCN^-$], of your unknown, with the actual [$SCN^-$]. Suggest reasons for the disparity.

3. (Part III) Use the net absorbance values, along with the best fit line equation of the standard solutions in Part I to determine the [$FeSCN^{2+}$] at equilibrium for each of the mixtures that you prepared in Part III. Complete the table below and give an example of your calculations.

| Test tube number | 2 | 3 | 4 | 5 |
|---|---|---|---|---|
| [$FeSCN^{2+}$] | | | | |

4. (Part III) Calculate the equilibrium concentrations for $Fe^{3+}$ and $SCN^-$ for the mixtures in test tubes 2-5 in Part III. Complete the table below and give an example of your calculations.

| Test tube number | 2 | 3 | 4 | 5 |
|---|---|---|---|---|
| $[Fe^{3+}]$ | | | | |
| $[SCN^-]$ | | | | |

5. Calculate the value of $K_{eq}$ for the reaction. Explain how you used the data to calculate $K_{eq}$.

**LABORATORY REPORT (90)**

Include your objective and *paraphrased* procedure. All data should be easily read…you might reproduce the tables that also include some of your calculated values for ease of reading. Address all five steps of the Data Analysis. Show one calculation of each type. Report the results of the remaining calculations. Remember to write your conclusions. I will give you the actual value of the $K_{eq}$ when I pass your report back to you. There will be no percent error calculation for this experiment.

# THE STANDARDIZATION OF SODIUM HYDROXIDE

Duration: 1 day

**INTRODUCTION**

The technique of titration finds many applications, but is especially useful in the analysis of acidic and basic substances. Titration involves measuring the exact volume of a solution of known concentration that is required to react with a measured volume of a solution of unknown concentration, or with a weighed sample of unknown solid. A solution of accurately known concentration is called a **standard solution**. Typically, to be considered a standard solution, the concentration of the solute in the solution must be known to four significant figures.

In many cases (especially with solid solutes) it is possible to prepare a standard solution by accurate weighing of the solute, followed by precise dilution to an exactly known volume in a volumetric flask. Such a standard is said to have been prepared determinately. One of the most common standard solutions used in analyses, however, cannot be prepared in this manner.

Solutions of sodium hydroxide are commonly used in titration analyses of samples containing an acidic solute. Although sodium hydroxide is a solid, it is not possible to prepare standard sodium hydroxide solutions by weight. Solid NaOH is usually of questionable purity. NaOH reacts with $CO_2$ from the atmosphere and is also capable of reacting with the glass of the container in which it is provided. For these reasons, NaOH solutions are generally prepared to be approximately a given concentration. They are then standardized by titration of a weighed sample of a primary standard acidic substance. By measuring how many milliliters of the approximately prepared NaOH are necessary to react completely with a weighed sample of a known primary standard acidic substance (in this case, potassium hydrogen phthalate, KHP), the concentration of the NaOH solution can be calculated. Once prepared, however, the concentration of NaOH solution will change with time (for the same reasons outlined earlier). As a consequence, NaOH solutions must be used relatively quickly.

In titration analyses, there must be some means of knowing when enough titrant has been added to react exactly and completely with the sample being titrated. In an acid/base titration analysis, there should be an abrupt change in pH when the reaction is complete. For example, if the sample being titrated is an acid, then the titrant to be used will be basic (probably NaOH). When one excess drop of titrant is added (beyond that needed to react with the acid sample), the solution being titrated will suddenly become basic. There are various natural and synthetic dyes, called indicators that exist in different colored forms at different pH values. A suitable indicator can be chosen that will change color at a pH value consistent with the point at which the titration reaction is complete. The indicator to be used in this experiment is phenolphthalein, which is colorless in acid solutions, but changes to a pink form at basic pH.

**OBJECTIVES**

In this experiment, you will

- Accurately standardize a solution.
- Become proficient in the use of acid-base titration.
- Determine the equivalence point of a weak acid-strong base titration.
- Calculate the molar concentration of a strong base solution.

**MATERIALS**

Sodium hydroxide,NaOH, pellets
Balance
250-mL volumetric flask
Potassium hydrogen phthalate, KHP
Beakers
Erlenmeyer flasks
Phenolphthalein solution
Wash bottle
Distilled water

**PROCEDURE**

1. Clean and rinse a 250-mL volumetric flask with several washings of tap water and then a final small quantity of distilled water. Label the flask "*Approximately 0.2 M NaOH*." Put about 100 mL of distilled water into the flask.

2. Weigh approximately 2 g of NaOH pellets on weighing paper and transfer to the 250-mL flask. Stopper and shake the flask to dissolve the NaOH pellets completely. When the NaOH pellets have completely dissolved, add distilled water to the bottle until the water level is at the etched line on the neck. Stopper and shake thoroughly to mix.

3. This NaOH is the titrant for the analyses that follow. Keep the bottle tightly stoppered when not actually in use (to avoid exposure to the air).

4. Set up a buret in a buret clamp. Rinse it with tap water, distilled water, and finally, a small portion of the NaOH that you just prepared. Then fill the buret with the NaOH and zero it, discarding any waste NaOH in a waste beaker.

5. Take a clean, dry beaker to the oven that contains the primary standard grade potassium hydrogen phthalate (KHP) in a 250-mL beaker. Using beaker tongs, remove the KHP from the oven. Place 3-4 spatulas of KHP into your beaker. Return the 250-mL beaker of KHP to the oven, and take your beaker to your lab station.

6. Allow the KHP to cool to room temperature. While it is cooling, clean and dry three 250-mL Erlenmeyer flasks. Label the flasks as 1, 2, and 3.

7. When the KHP is completely cool, weigh three samples of KHP on the analytical balance between 0.6 and 0.8 g, one for each of the Erlenmeyer flasks. Record the exact mass to 0.0001g of each KHP sample in each flask.

8. Add 100 mL of distilled water to flask #1. Add 2-3 drops of phenolphthalein and swirl to dissolve the KHP sample completely.

9. With a white sheet of paper or paper towel beneath the flask, begin adding NaOH solution from the buret to the sample in flask #1, swirling the flask constantly during the addition. As the flashes of pink persist longer and longer, begin adding the NaOH one drop at a time, with constant swirling. You will stop titrating when one drop makes the pink color persist for at least 30 seconds with constant agitation. Record the volume of NaOH used to the nearest 0.01 of a mL.

10. Repeat the titration for the remaining two flasks, recording the volume of NaOH required for each.

11. Keep your NaOH solution tightly sealed in preparation for the next experiment. Dispose of the solutions in the Erlenmeyer flasks as indicated.

**DATA ANALYSIS**

1. Using the mass of KHP, the molar mass of KHP, the stoichiometric ratio (1:1), the volume of NaOH used, calculate the molarity of the NaOH solution for each trial. Average the molarity.

**LABORATORY REPORT (25)**

Include your objective and *paraphrased* procedure. All data should be easily read. Show one calculation of each type. Report all calculation results. Remember to write your conclusions. You will use your standardized solution of NaOH and its average molarity for the next experiment.

# ACID-BASE TITRATION

Duration: 2 days

**INTRODUCTION**

A titration is a process used to determine the volume of a solution that is needed to react with a given amount of another substance. In this experiment, your goal is to determine the molar concentration of two acid solutions by conducting titrations with a base of known concentration. You will be testing a strong acid, HCl, solution and a weak acid, $HC_2H_3O_2$, solution. You will use the sodium hydroxide, NaOH, solution that you standardized in Lab 6 as your base of known concentration. The reaction equations are shown below in net ionic form.

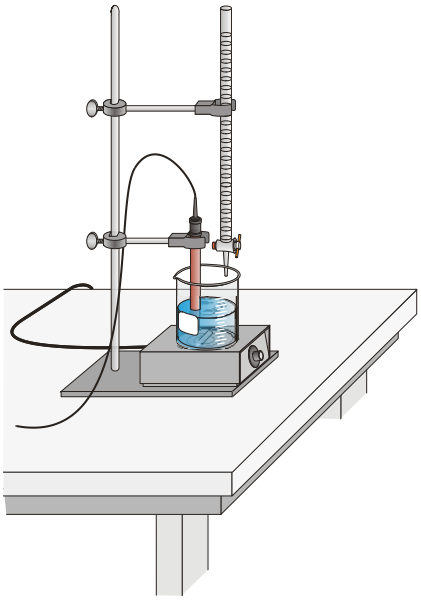
$$H^+ (aq) + OH^- (aq) \rightarrow H_2O(l)$$

$$HC_2H_3O_2(aq) + OH^- (aq) \rightarrow H_2O(l) + C_2H_3O_2^- (aq)$$

The stoichiometry of the two reactions is identical; thus, your calculations will be straightforward. However, you will observe a significant difference in how the two acid solutions react with NaOH. In this experiment, you will use a handheld device to monitor pH as you titrate. The region of most rapid pH change will then be used to determine the equivalence point. The volume of NaOH titrant used at the equivalence point will be used to determine the molarity of the acid solutions.

**OBJECTIVES**

- In this experiment, you will
  - Accurately conduct acid-base titrations.
  - Determine the equivalence point of a strong acid-strong base titration.
  - Determine the equivalence point of a weak acid-strong base titration.
  - Calculate the molar concentrations of two acid solutions.



*Titration Apparatus*

**MATERIALS**

Vernier LabQuest
Vernier pH Sensor
0.100 M NaOH solution
Hydrochloric acid, HCl, solution, unknown molarity
Acetic acid, $HC_2H_3O_2$, solution, unknown molarity
Magnetic stirrer
Stirring bar
Wash bottle
Distilled water
Ring stand/clamps/burets
Beaker
Graduated cylinder

**PROCEDURE**

1. Add 50 mL of distilled water to a beaker. Use a buret to transfer 10.0 mL of the HCl solution into the beaker. **CAUTION:** *Handle the hydrochloric acid with care. It can cause painful burns if it comes in contact with the skin*.

2. Place the beaker on a magnetic stirrer and add a stirring bar. If no magnetic stirrer is available, stir the reaction mixture with a stirring rod during the titration.

3. Connect the pH Sensor to LabQuest and choose New from the File menu.

4. Set up a ring stand, buret clamp, and buret to conduct the titration (see Titration Apparatus). Rinse and fill the buret with 0.100 M NaOH solution. **CAUTION:** *Sodium hydroxide solution is caustic. Avoid spilling it on your skin or clothing*.

5. Use a clamp to suspend the pH Sensor on the ring stand, as shown in Titration Apparatus. Position the pH Sensor so that its tip is immersed in the HCl solution but is not struck by the stirring bar. Gently stir the beaker of acid solution.

6. On the Meter screen, tap Mode. Change the data-collection mode to Events with Entry. Enter the Name (Volume) and Unit (mL) and select OK.

7. Conduct the titration carefully, as described below.
   - Start data collection.
   - Before you have added any NaOH solution, tap Keep and enter **0** as the buret volume in mL. Select OK to store the first data pair.
   - Add the next increment of NaOH titrant (enough to raise the pH about 0.15 units). When the pH stabilizes, tap Keep, and enter the current buret reading as precisely as possible. Select OK to save the second data pair.
   - Continue adding NaOH solution in increments that raise the pH by about 0.15 units and enter the buret reading after each increment. When a pH value of approximately 3.5 is reached, change to a one-drop increment. Enter a new buret reading after each increment.
   - After a pH value of approximately 10 is reached, again add larger increments that raise the pH by about 0.15 pH units, and enter the buret level after each increment.
   - Continue adding NaOH solution until the pH value remains constant.

8. Stop data collection to view a graph of pH *vs.* volume.

9. Dispose of the reaction mixture as directed. Rinse the pH Sensor with distilled water in preparation for the second titration.

10. Examine your titration data to identify the region where the pH made the greatest increase. The equivalence point is in this region.
    - To examine the data pairs on the displayed graph, select any data point.
    - As you move the examine line, the pH and volume values of each data point are displayed to the right of the graph.
    - Identify the equivalence point as precisely as possible and record this information.
    - Store the data from the first run by tapping the File Cabinet icon and store on a flash drive.
11. An alternate way of determining the precise equivalence point of the titration is to take the first and second derivatives of the pH-volume data.

    Determine the peak value on the first derivative *vs.* volume plot.
    - Tap the Table tab and choose New Calculated Column from the Table menu.
    - Enter d1 as the Calculated Column Name. Select the equation 1st Derivative (Y,X). Use Volume as the Column for X and pH as the Column for Y. Select OK.
    - On the displayed plot of d1 *vs.* volume, examine the graph to determine the volume at the peak value of the first derivative.

    Determine the zero value on the second derivative *vs.* volume plot.

    d. Tap Table and choose New Calculated Column from the Table menu.
    e. Enter d2 as the Calculated Column Name. Select the equation 2nd Derivative (Y,X). Use Volume as the Column for X and pH as the Column for Y. Select OK.
    f. On the displayed plot of d2 *vs.* volume, examine the graph to determine the volume when the 2nd derivative equals approximately zero.
12. Putting aside the ph meter and using bromothymol blue as an indicator, repeat the titration with a second HCl solution. Bromothymol blue will reach a green endpoint at the equivalence point.

13. Using the pH meter, conduct two titration trials with the $HC_2H_3O_2$ solution. Note that the equivalence point of this titration will not be identical to the HCl titration. Analyze the titration results and record the equivalence point in your data table.

14. Discard the drained NaOH solution in the 250 mL beaker as directed.

**DATA TABLE**

| HCl Trial | Volume HCl (mL) | Volume NaOH (mL) |
|---|---|---|
| 1 | | |
| 2 | | |

| $HC_2H_3O_2$ Trial | Volume $HC_2H_3O_2$ (mL) | Volume NaOH (mL) |
|---|---|---|
| 1 | | |
| 2 | | |

**DATA ANALYSIS**

1. Using the molarity of the NaOH, its required volume to reach endpoint, the stoichiometric ratio, and the volume of acid used, calculate the molarity of the HCl solution and the $HC_2H_3O_2$ solution for each trial.

2. The equivalence points of the titrations curves for the HCl solution and the $HC_2H_3O_2$ solution were not in the same pH range. Explain.

**LABORATORY REPORT (35)**

Include your objective and *paraphrased* procedure. All data should be easily read. Show one calculation of each type. Average your molarities for each of the acids. Report all calculation results. Address both Data Analysis steps. Remember to write your conclusions. You will be graded in part on your accuracy of the molarities. I will give you the actual values when I pass your report back to you. At that time, you should add a calculation of your percentage errors in your lab notebook.

# BUFFERS

Duration: 2 days

**INTRODUCTION**

A buffer is a mixture of a weak acid and its conjugate base, or a weak base and its conjugate acid. A buffer's function is to absorb acids ($H^+$ or $H_3O^+$ ions) or bases ($OH^-$ ions) so that the pH of the system changes very, very little.

In many systems, buffers are critical. Blood plasma, a natural example in humans, is a bicarbonate buffer that keeps the pH of blood between 7.2 and 7.6.

By design, a buffer is an equilibrium system. For example, a buffer can be prepared with nitrous acid, $HNO_2$. The weak acid establishes an aqueous equilibrium as shown below.

$$HNO_2\ (aq) \leftrightarrow H^+\ (aq) + NO_2^-\ (aq)$$

The equilibrium constant expression is shown below.

$$K_a = \frac{[H^+][NO_2^-]}{[HNO_2]}$$

To prepare a buffer system with nitrous acid, a conjugate base is added, such as sodium nitrite ($NaNO_2$). The resulting system is a mixture of $HNO_2$ and $NO_2^-$ ions. The nitrous acid molecule will neutralize hydroxide ions and the nitrite ion from the conjugate will neutralize hydrogen ions.

A variation of the equilibrium expression above, called the Henderson-Hasselbalch equation, is the best reference in preparing a buffer solution. For our nitrous acid/sodium nitrate buffer example, the Henderson-Hasselbalch equation is shown below.

$$pH = pK_a + \log\frac{[NO_2^-]}{[HNO_2]}$$

The pH range in which a buffer solution is effective is generally considered to be ±1 of the $pK_a$.

In this experiment, you will use the Henderson-Hasselbalch equation to determine the amount of acetic acid and sodium acetate needed to prepare two acidic buffer solutions. You will then prepare the buffers and test their buffer capacities by adding solutions of NaOH and HCl.

**OBJECTIVES**

In this experiment, you will

- Prepare and test two acid buffer solutions.
- Determine the buffer capacity of the prepared buffers.

**MATERIALS**

Vernier LabQuest
Vernier pH Sensor
Magnetic stirrer and stirring bar
Beakers
Graduated cylinders
Ring stand/clamps/burets
Balance

0.5 M sodium hydroxide, NaOH, solution
0.5 M hydrochloric acid, HCl, solution
0.1 M acetic acid, $HC_2H_3O_2$, solution
1.0 M acetic acid, $HC_2H_3O_2$, solution
Solid sodium acetate, $NaC_2H_3O_2$
Distilled water

**PRE-LAB EXERCISE**

Use the Henderson-Hasselbalch equation to perform the following calculations. The $K_a$ of acetic acid is $1.8 \times 10^{-5}$. Check your calculations with me before preparing the buffer solutions.

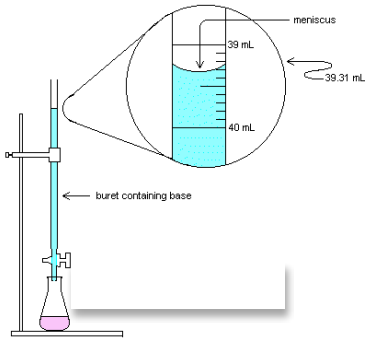
- Buffer A: Calculate the mass of solid sodium acetate required to mix with 100.0 mL of 0.1 M acetic acid to prepare a pH 4 buffer. Record the mass in your data table.
- Buffer B: Calculate the mass of solid sodium acetate required to mix with 100.0 mL of 1.0 M acetic acid to prepare a pH 4 buffer. Record the mass in your data table.

**PROCEDURE**

**Part I Prepare and Test Buffer Solution A**

1. Use your calculations from the Pre-Lab Exercise to prepare 100 mL of Buffer A. Weigh out the precise mass of sodium acetate and dissolve it in 100.0 mL of 0.1 M acetic acid solution.

2. Set up two burets, buret clamp, and ring stand (see Titration Apparatus). Rinse and fill one buret with 0.5 M NaOH solution. Rinse and fill the second buret with 0.5 M HCl solution. **CAUTION:** *Sodium hydroxide solution is caustic. Avoid spilling it on your skin or clothing. Handle the hydrochloric acid with care. It can cause painful burns if it comes in contact with the skin.*




*Titration Apparatus*

4. Use a graduated cylinder to measure out 10.0 mL of the Buffer A solution into a 250 mL beaker and add 15 mL of distilled water. Place the beaker on a magnetic stirrer, beneath the buret of NaOH, and add a stirring bar. If no magnetic stirrer is available, you will stir with a stirring rod during the testing.

5. Connect the pH Sensor to LabQuest and choose New from the File menu. Suspend the pH Sensor in the pH 4 buffer solution, as shown. Make sure that the sensor is not struck by the stirring bar.

6. On the Meter screen, tap Mode. Change the data-collection mode to Events with Entry. Enter the Name (Volume) and Units (mL). Select OK.
7. Collect data. You will slowly and carefully add 0.50 M NaOH solution to the sample of Buffer A.
   - Start data collection.
   - Take an initial pH reading of the buffer solution. Allow the pH readings to stabilize and then tap Keep. Enter **0** as the buret volume in mL. Select OK to save the first data pair. Record the initial pH value in your data table.
   - Add a small amount of the NaOH solution, up to 0.50 mL. When the pH stabilizes, tap Keep. Enter the current buret reading, and then select OK to save the second data pair.
   - Continue adding the NaOH solution in small increments that raise the pH consistently and enter the buret reading after each increment. Your goal is to raise the pH of the buffer by precisely 2 pH units.
   - When the pH of the buffer solution is precisely 2 units greater than the initial reading, continue to add the NaOH solution in small increments until you have reached, and passed, the equivalence point of the titration.
   - Stop data collection to view the graph.
8. Examine your titration curve.
   - To examine the data pairs on the displayed graph, select any data point. As you tap each point, the pH and volume values of each data point are displayed to the right of the graph.
   - Identify the point at which the pH increased by 2 units and record the volume of NaOH in your data table.
   - Store the data from the first trial by tapping the File Cabinet icon and storing on a flash drive.
9. Dispose of the reaction mixture as directed. Rinse the pH Sensor with distilled water in preparation for the second trial.
10. Repeat Steps 7–9, using a fresh 10.0 mL sample of the Buffer A solution. For this second trial, titrate the buffer with 0.5 M HCl solution. Carefully add HCl in small increments until the pH of the solution has been lowered by precisely 2 units. Record, in your data table, the volume of HCl that was used. You may save the data if you wish.

**Part II Prepare and Test Buffer Solution B**

11. Use your calculations from the Pre-Lab Exercise to prepare 100 mL of Buffer B. Weigh out the precise mass of sodium acetate and dissolve it in 100.0 mL of 1.0 M acetic acid solution. If necessary, refill the burets of NaOH and HCl solution.
12. Use a graduated cylinder to measure out 10.0 mL of the Buffer B solution and add 15 mL of distilled water. Repeat the necessary steps to test Buffer B in a manner similar to the Part I trials. Determine the volumes of NaOH and HCl that were used to change the pH of Buffer B by 2 units and record the values in your data table.

**DATA TABLE**

| | Buffer A | Buffer B |
|---|---|---|
| Mass of $NaC_2H_3O_2$ used to prepare buffer (g) | | |
| Volume of buffer prepared (mL) | 100.0 | 100.0 |
| Molar concentration of $HC_2H_3O_2$ in buffer (M) | 0.1 | 1.0 |
| Initial pH of buffer | | |
| Volume of 0.5 M NaOH to raise pH by 2 units (mL) | | |
| Volume of 0.5 M HCl to lower pH by 2 units (mL) | | |
| Volume of 0.5 M NaOH at equivalence point (mL) | | |

**DATA ANALYSIS**

1. Write reactions to explain how your acetic acid-acetate buffer reacts with a base and how it reacts with an acid.

2. Graph the average volume of NaOH required for each $HC_2H_3O_2$ solution *vs* the average pH values.

3. Buffer capacity has a rather loose definition, yet it is an important property of buffers. A commonly seen definition of buffer capacity is "the amount of hydrogen ion or hydroxide ion than can be neutralized by a buffer before the pH changes significantly." Use your data to determine the buffer capacities of Buffers A and B.

**LABORATORY REPORT (35)**

Include your objective and *paraphrased* procedure. All data should be easily read. Show one calculation of each type from your pre-lab exercises. Report all calculation results. Address all three steps of Data Analysis in your report. Remember to write your conclusions.

# DETERMINING A SOLUBILITY PRODUCT CONSTANT

Duration: 2 days

**INTRODUCTION**

The solubility product constant, $K_{sp}$, is a particular type of equilibrium constant. The equilibrium is formed when an ionic solid dissolves in water to form a saturated solution. The equilibrium exists between the aqueous ions and the undissolved solid. A saturated solution contains the maximum concentration of ions of the substance that can dissolve at the solution's temperature. The equilibrium equation showing the ionic solid lead chloride dissolving in water is:

$$PbCl_2(s) \Leftrightarrow Pb^{2+}(aq) + 2Cl^-(aq)$$

The solubility product expression is:

$$K_{sp} = [Pb^{2+}][Cl^-]^2$$

where square brackets refer to molar concentrations of the ions. A knowledge of the $K_{sp}$ of a salt is useful, since it allows you to determine the concentration of ions of the compound in a saturated solution. This allows you to control a solution so that precipitation of a compound will not occur, or to find the concentration needed to cause a precipitate to form.

The solubility product which will be determined by this experiment is that of calcium hydroxide.

**OBJECTIVES**

In this experiment, you will

- Perform serial dilutions.
- Determine solubility product constant o calcium hydroxide.

**MATERIALS**

| | |
|---|---|
| Beakers | 0.1 M sodium hydroxide, NaOH, solution |
| 96-well microplate | 0.1 M calcium nitrate, $Ca(NO_3)_2$, solution |
| Beral plastic pipets | Distilled water |

**PROCEDURE**

1. Arrange a microplate so that you have 12 wells across from left to right. Put 5 drops of 0.10 M calcium nitrate in well #1 in the first row.

2. Place 5 drops of water in each of the wells #2 through #12 in the first row.

3. Next, add 5 drops of 0.10 M calcium nitrate to well #2. Use an empty Beral pipet to mix the solution thoroughly by drawing the solution into the pipet and then squirting it back several times. The solution in this well, #2, is now 0.050 M in $Ca^{2+}$ ion.

4. Use your empty pipet to remove the solution from well #2 and put 5 drops of this solution into well #3. Put the remaining solution back in well #2. Mix the solution in well #3 as before.

5. Continue this serial dilution procedure, adding 5 drops of the previous solution to the 5 drops of water in each well down the row until you fill the last one, #12. Mix the solution in well #12, and discard 5 drops.

6. Determine the concentration of solution in each well, and verify that the concentration of calcium ions in well #12 is 4.9 x 10-5 M.

7. Place 5 drops of 0.10 M sodium hydroxide, NaOH, in each of the wells #1 through #12. When the sodium hydroxide is added to each well, the initial concentrations of the reactants are halved, as each solution dilutes the other. Use 12 empty pipets to mix each of these combined solutions by drawing each solution up into the pipet and squirting it back into the well; or mix thoroughly with a toothpick. Now the concentration of $Ca^{2+}$ ions in well #12 is 2.4 x 10-5 M.

8. Allow three or four minutes for the precipitates to form. Observe the pattern of precipitation. At one point the concentration of both ions becomes too low to have any precipitate form. You will assume that the first well with no precipitate represents a saturated solution.

9. To check your results, repeat the procedure but use a serial dilution of the NaOH. In a different row, put 5 drops of 0.10 M sodium hydroxide in well #1.

11. Put 5 drops of distilled water in wells #2 through # 12. Add 5 drops of the 0.10 M sodium hydroxide to well #2. Use an empty Beral pipet to mix the solution by pulling the solution into the pipet and then squirting it back several times. The solution in this well, #2, is now 0.050 M in $OH^-$ ion.

12. Continue the serial dilution to well #12, and then remove 5 drops from well #12.

13. Add 5 drops of 0.10 M $Ca(NO_3)_2$ to each of the wells, and mix each with an empty pipet or toothpick. Again, determine the well where no more precipitate appears.

14. Calculate the concentration of calcium and hydroxide ions in the first well where there is no precipitate, and again, calculate the value of $K_{sp}$.

**DATA ANALYSIS**

1. Using the data from steps 1-8, calculate the concentration of $Ca^{2+}$ ions and $OH^-$ ions in the well where no more precipitate appears. Using these concentrations, determine the solubility product, the $K_{sp}$ of calcium hydroxide.

2. Using the data from steps 9-13, calculate the concentration of $Ca^{2+}$ ions and $OH^-$ ions in the well where no more precipitate appears. Using these concentrations, again determine the solubility product, the $K_{sp}$ of calcium hydroxide.

**LABORATORY REPORT (35)**

Include your objective and *paraphrased* procedure. Your observations of each step should be clear. You need only show one calculation of each type. Show the results of all of your calculations in your report. Address both steps in Data Analysis. Remember to write your conclusions. You will be graded in part on your accuracy. I will give you the actual $K_{sp}$ when I pass your report back to you. At that time, you should add a calculation of your percentage error in your lab notebook.

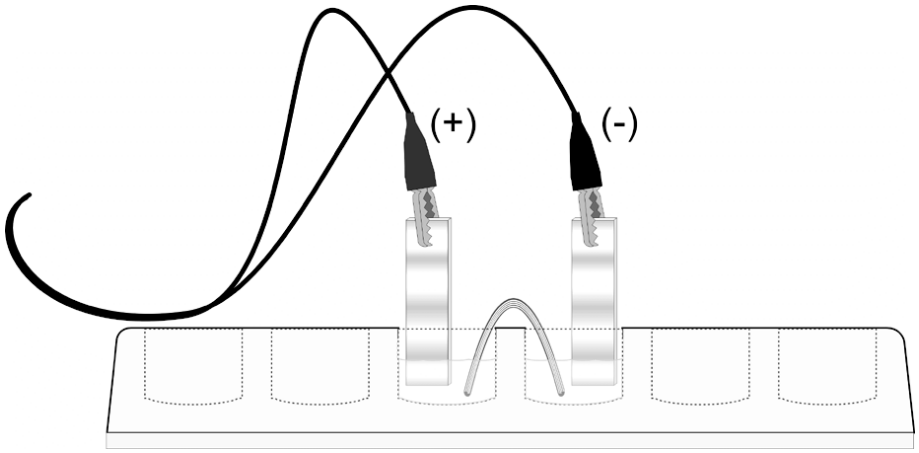
# ELECTROCHEMISTRY: VOLTAIC CELLS

Duration: 2 days

**INTRODUCTION**

In electrochemistry, a voltaic cell is a specially prepared system in which an oxidation-reduction reaction occurs spontaneously. This spontaneous reaction produces an easily measured electrical potential. Voltaic cells have a variety of uses.

In this experiment, you will prepare a variety of semi-microscale voltaic cells in a 24-well test plate. A voltaic cell is constructed by using two metal electrodes and solutions of their respective salts (the electrolyte component of the cell) with known molar concentrations. In Parts I and II of this experiment, you will use a Voltage Probe to measure the potential of a voltaic cell with copper and lead electrodes. You will then test two voltaic cells that have unknown metal electrodes and, through careful measurements of the cell potentials, identify the unknown metals. In Part III of the experiment, you will measure the potential of a special type of voltaic cell called a concentration cell. In the first concentration cell, you will observe how a voltaic cell can maintain a spontaneous redox reaction with identical copper metal electrodes, but different electrolyte concentrations. You will then measure the potential of a second concentration cell and use the Nernst equation to calculate the solubility product constant, $K_{sp}$, for lead iodide, $PbI_2$.




*Voltaic Cells*

**OBJECTIVES**

In this experiment, you will

- Prepare a Cu-Pb voltaic cell and measure its potential.
- Test two voltaic cells that use unknown metal electrodes and identify the metals.
- Prepare a copper concentration cell, observe, and measure its potential.
- Prepare a lead concentration cell and measure its potential.
- Use the Nernst equation to calculate the $K_{sp}$ of $PbI_2$.

**MATERIALS**

Vernier LabQuest
Vernier Voltage Probe
Graduated cylinders
24-well test plate
String
Cu and Pb electrodes
Two unknown electrodes, labeled X and Y
Beakers
Plastic Beral pipets
0.10 M copper (II) nitrate, $Cu(NO_3)_2$, solution
0.10 M lead (II) nitrate, $Pb(NO_3)_2$, solution
1.0 M copper (II) sulfate, $CuSO_4$, solution
0.050 M potassium iodide, KI, solution
1 M potassium nitrate, $KNO_3$, solution
0.10 M X nitrate solution
0.1 M Y nitrate solution
Steel wool

**PRE-LAB EXERCISE**

Use the table of standard reduction potentials in your text, or reputable online source, to complete the following table. Remember that voltaic cells are to made. An example is provided.

| Electrodes | Half-reactions | $E^\circ$ | $E^\circ_{cell}$ |
|---|---|---|---|
| Zn<br>Cu | $Zn(s) \rightarrow Zn^{2+} + 2e^-$<br>$Cu^{2+} + 2e^- \rightarrow Cu(s)$ | +0.76 V<br>+0.34 V | +1.10 V |
| Cu<br>Pb | | | |
| Pb<br>Ag | | | |
| Pb<br>Mg | | | |
| Pb<br>Zn | | | |

**PROCEDURE**

**Part I Determine the $E^o$ for a Cu-Pb Voltaic Cell**

1. Use a 24-well test plate as your voltaic cell. Use Beral pipets to transfer small amounts of 0.10 M $Cu(NO_3)_2$ and 0.10 M $Pb(NO_3)_2$ solution to two neighboring wells in the test plate. CAUTION: *Handle these solutions with care.*

2. Obtain one Cu and one Pb metal strip to act as electrodes. Polish each strip with steel wool. Place the Cu strip in the well of $Cu(NO_3)_2$ solution and place the Pb strip in the well of $Pb(NO)_3$ solution. These are the half cells of your Cu-Pb voltaic cell.

3. Make a salt bridge by soaking a short length of string in a beaker than contains a small amount of 1 M $KNO_3$ solution. Connect the Cu and Pb half cells with the string.

4. Connect the Voltage Probe to LabQuest and choose New from the File menu.

5. Measure the potential of the Cu-Pb voltaic cell. Complete the steps quickly to get the best data.
   - You will read the potential, in volts, on the LabQuest screen. Write down your readings in your notebook.
   - Connect the leads from the Voltage Probe to the Cu and Pb electrodes to get a positive potential reading. Record the potential immediately after making the connection with the Voltage Probe.
   - Remove both electrodes from the solutions. Clean and polish each electrode.
   - Put the Cu and Pb electrodes back in place to set up the voltaic cell. Connect the Voltage Probe, as before. Record the potential immediately after making the connection with the Voltage Probe.
   - Remove the electrodes. Clean and polish each electrode again.
   - Set up the voltaic cell a third, and final, time. Record the potential immediately after making the connection with the Voltage Probe.
   - Calculate the mean of your three readings and record it in your data table as the average potential for the Cu-Pb voltaic cell.

**'Part II Determine the $E^o$ for Two Voltaic Cells Using Pb and Unknown Metals**

6. Obtain a small amount of the unknown electrolyte solution labeled "0.10 M X" and the corresponding metal strip, X.

7. Use a Beral pipet to transfer a small amount of 0.10 M X solution to a well adjacent to the 0.10 M $Pb(NO_3)_2$ solution in the test plate.

8. Make a new salt bridge by soaking a short length of string in the beaker of 1 M $KNO_3$ solution. Connect the X and Pb half cells with the string.

9. Measure the potential of the X-Pb voltaic cell as you did for the Cu-Pb voltaic cell in step 5, substituting X for Cu.

10. Repeat Steps 6–9 using the unknown and its corresponding electrolyte solution labeled "Y".

**Part III Prepare and Test Two Concentration Cells**

11. Set up and test a copper concentration cell.
    - Prepare 20 mL of 0.050 M $CuSO_4$ solution by mixing 1 mL of 1.0 M $CuSO_4$ solution with 19 mL of distilled water.
    - Set up a concentration cell in two wells of the 24-well test plate by adding 5 mL of 0.050 M $CuSO_4$ solution to one well and 5 mL of 1.0 M $CuSO_4$ solution to an adjacent well. Use Cu metal electrodes in each well. Use a $KNO_3$-soaked string as the salt bridge, as in Parts I and II.
    - Test and record the potential of the concentration cell in the same manner that you tested the voltaic cells in Parts I and II.

12. Determine the solubility product constant, $K_{sp}$, of $PbI_2$.
    - Prepare a small amount of 0.050 M $Pb(NO_3)_2$ solution by diluting 0.10 M $Pb(NO_3)_2$ solution.
    - Mix 9 mL of 0.050 M KI solution with 3 mL of 0.050 M $Pb(NO_3)_2$ solution in a small beaker.
    - Set up the half cells in neighboring wells of the 24-well test plate. Place 5 mL of 0.050 M $Pb(NO_3)_2$ solution in one half cell, and 5 mL of the $PbI_2$ mixture, from the small beaker, into an adjacent half cell. Use Pb electrodes in each half cell. Use a $KNO_3$-soaked string as the salt bridge.
    - Test and record the potential of the cell in the same manner that you tested the voltaic cells in Part I.
13. Discard the electrodes and the electrolyte solutions as directed. Rinse and clean the 24-well plate.

**DATA TABLE**

| *Results of Parts I and II* | Cu/Pb | X/Pb | Y/Pb |
|---|---|---|---|
| Average cell potential (V) | | | |

| *Results of Part III* | Cu concentration | $Pb/PbI_2$ |
|---|---|---|
| Average cell potential (V) | | |

**DATA ANALYSIS**

1. (Part I) Compare the average cell potential, for your Cu/Pb cell, with the $E^\circ_{cell}$ that you calculated in the pre-lab exercise. Explain why your cell potential is different from the cited value.

2. (Part II) The unknown metals X and Y were either magnesium, silver, or zinc. Use the cited value for the reduction potential of Pb and the measured cell potential for the unknowns to identify X and Y.

3. (Part III) Use the Nernst equation to calculate the theoretical value of *E* of the copper concentration cell and compare this value with the cell potential that you measured.

4. (Part III) Use the Nernst equation and the information that you collected about the $Pb/PbI_2$ cell to complete the following calculations:
   a. Use the cell potential for the $Pb/PbI_2$ cell and the known $[Pb^{2+}]$ to calculate the $[Pb^{2+}]$ in equilibrium with $PbI_2$.
   b. Use the original diluted $[Pb^{2+}]$ and $[I^-]$ to calculate the $[I^-]$ in solution.
   c. Use your data to calculate the $K_{sp}$ of $PbI_2$.
   d. Compare your experimental value to the accepted value of $K_{sp}$ of $PbI_2$ as $9.8 \times 10^{-9}$.

**LABORATORY REPORT (40)**

Include your objective and *paraphrased* procedure. All data should be easily read. Report all calculation results. Address all four steps of Data Analysis in your report. Remember to write your conclusions.

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