# Experiment 1 – Separation of Copper(II) Sulfate from Sand

#### **Discussion**

Mixtures are a combination of substances in which the components keep their individual characteristics. Mixtures have variable proportions and can be separated by simple physical means. The mixture's components have different physical properties like melting point, boiling point, or solubility that allow us to selectively remove individual components from the mixture. Once separated, the percentage of each component in the original mixture can be calculated.

In this experiment, you will separate a mixture of copper(II) sulfate and sand using the physical property of solubility. You will learn about certain methods of separation that include decantation, filtration, and evaporation. Finally, the Law of Conservation of Mass will be applied to check the validity of your final calculations.

#### **Procedure**

- 1. Weigh about 4 –5 grams of the CuSO<sub>4</sub>/sand mixture in a 100 mL beaker on the laboratory balance by taring (your instructor will explain and demonstrate).
- 2. Add 10–15 mL of D.I. water to the beaker, and swirl. Next, weigh and record the weight of a piece of filter paper AND an evaporating dish separately. Then assemble the filter apparatus as demonstrated by the instructor, filter the mixture, and collect the filtrate (liquid) onto the evaporating dish. Use your wash bottle (filled with D.I. water) to transfer all the undissolved solid from the beaker to the filter paper. After all the liquid has drained through the filter, wash the filter with small portions of D.I. water from the wash bottle until the washings are colorless. Again, make sure you collect ALL the filtrate and washings in the evaporating dish. Try to use small amounts of water. You will be evaporating the liquid; the more water you add, the longer it will take!
- 3. Prepare a steam bath by placing a 250 mL beaker, 2/3 full of water with 4 5 boiling chips, on a wire screen on a ring stand. Place the evaporating dish carefully on the beaker and heat the water to boiling. Heat the steam bath until the filtrate has completely evaporated. Do NOT let the steam bath boil to dryness; you may have to refill the water in the beaker occasionally. If the beaker goes dry, it must be cooled before adding water to prevent the beaker from cracking!
- 4. Open the filter paper onto a large watch glass that has been labeled with your drawer number using a permanent marker. Dry the sand on the filter paper/watch glass in the drying oven, set at 150 °C, for 20 minutes. Afterwards, carefully remove the watch glass, allow it to cool, and weigh the filter paper with the sand by placing it into a TARED weighing boat on the analytical balance. Calculate the mass of your sand.
- 5. Once your filtrate has completely evaporated and your salt remains on the evaporating dish, weigh the evaporating dish with the salt sample. Compute the weight of the CuSO<sub>4</sub> sample by difference.
- 6. Calculate the weight percent CuSO<sub>4</sub> and sand in the sample.

Name:	Section:	
Data and Calculations for Experiment 1		
Mass of CuSO <sub>4</sub> /sand mixture		
Mass of empty evaporating dish		
Mass of evaporating dish and dry CuSO <sub>4</sub>		
Mass of CuSO <sub>4</sub>		
Mass of filter paper		
Mass of filter paper and sand		
Mass of sand		
Widss of Salid		
Total mass of recovered sand and CuSO <sub>4</sub>		
Calculated total percent recovery		
Percent by mass of $CuSO$		
refective by mass of Custo4.		
Show Calculation		
Percent by mass of sand:		
Show Calculation		

Name: \_\_\_\_\_

#### Questions

1. Many students do NOT recover 100% of the original mixture. Describe at least TWO possible problems that could cause LESS than 100% recovery of the mixture.

2. A student obtained the following data:

Mass of beaker	25.87 g
Mass of beaker with mixture sample	28.12 g
Mass of evaporating dish	146.36 g
Mass of evaporating dish with dried salt	147.10 g
Mass of beaker with dried sand	???

However, this student spills her sand sample out of the evaporating dish before weighing it. If the student believes in the Law of Conservation of Mass, what should have been the weight of the beaker with the dried sand in it? Show all your work.

3. A student receives a sample of a mixture with three components: (1) solid iodine that is first removed from the mixture by evaporation, (2) solid salt that is dissolved to separate it from the third component, and (3) solid sand. The salt and sand are dried and weighed, but the iodine escapes as a gas and is not recovered. The student starts with 4.25 g of the mixture and recovers 1.16 g of salt and 2.40 g of sand. What is the percent of each component in the original mixture? Show all your work.

## Experiment 2 – Measurements

#### Discussion

Experimental sciences, such as Chemistry, depend on making and using measurements properly. The SI system of units (sometimes called the metric system) is used almost exclusively. This system is very similar to our monetary system: \$1 = 10 dimes = 100 cents = 1000 mils. In chemistry, the basic units of length, mass, and volume are the meter, gram, and liter, respectively. They all are divided the same way. For example, 1 meter = 10 decimeters = 100 centimeters = 1000 millimeters. The kilo is also commonly used; it equals 1000 of the basic unit. For example, 1 kilogram = 1000 grams.

Often you will be asked to compare you experimental or calculated value to an "accepted" or theoretical value. The closer you are to the accepted value, the greater the accuracy of your experiment. The accepted value could be located in a reference such as the Handbook of Chemistry and Physics. Percent error is a common method for reporting accuracy, where a smaller percent error represents a more accurate experimental value. Percent error is calculated as:

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

In science, an experimenter is allowed to estimate one more digit past what can be measured exactly on an instrument. For example, if the smallest lines on a ruler are centimeters, and an object's length falls between 2 lines, more precision is gained by estimating between the lines. Therefore, the length of an object might be reported as 25.5 cm. The reported numbers are called "significant figures", and the more precise the instrument, the more significant figures it can produce.

A calculation cannot be any more precise than the least precise measurement. For example, density is calculated by dividing the mass of an object by its volume. Therefore, the density of an object might be 23.57 g/ 4.2 mL = 5.61190476 g/mL. But, the least precise measurement (the volume) only has a precision of 2 significant figures. Therefore, the density must be reported as 5.6 g/mL.

#### **Procedure**

Record your data on the report form as you complete the measurements.

#### A. Temperature

5 beakers with thermometers have been set up for you: (1) room temperature water, (2) boiling water, (3) a mixture of ice and water, (4) a stirred mixture of ice and water, and (5) a stirred mixture of ice, water and salt. Observe and record all temperatures to the nearest 0.1  $\degree$ C.

#### Name: \_

Section: \_

### B. Mass

When using any measuring device, never round off your raw data. If the balance fluctuates on the last digit, estimate that value. Weigh a (1) 100-mL beaker, (2) a 250-mL Erlenmeyer flask, (3) a plastic empty weighing boat, and (4) and then add approximately 2 grams of sodium chloride to the weighing boat. Calculate the mass of the sodium chloride added.

### C. Length

Using a metric ruler, measure the following in centimeters, remembering to estimate one extra digit: (1) the length of the double arrow on the report sheet, (2) the length of the external height of a 250-mL beaker, and (3) the length of a medium sized test tube.

#### D. Volume

The graduated cylinder is the most accurate equipment in your locker for measuring volume and can give a precision of 0.1 mL. Water is attracted to the glass sides of the cylinder, causing a curved effect called the meniscus. The cylinder should be read at eye level using the bottom of the meniscus. In theory, a 250-mL Erlenmeyer flask with a marking for 200 mL should have a volume of 200 mL at that mark! However the problem is that volumes marked on beakers and flasks are only approximate values. Therefore, fill a 250-mL Erlenmeyer flask to the <u>200 mL</u> mark with water, transfer this volume of water to a 250-mL graduated cylinder, and determine the exact volume.

It is often convenient to estimate volumes of 5 and 10 mL simply by observing the height of a liquid in a test tube. Use your graduated cylinder to place 5 and 10 mL of water in a medium-sized test tube and measure the heights in cm.

### E. Density

Density measures the "compactness" of material. For example, lead has a high density, and Styrofoam has a low density. Mathematically, this compactness is expressed as mass per unit volume. In chemistry, we use grams and milliliters: d = g/mL. Density is an intrinsic value; it does not depend on the amount of sample taken. We will take advantage of this by measuring the density of various sample sizes and averaging their densities:

- 1. Obtain 5 pieces of the same object and record its name on the data sheet.
- 2. Weigh each object and record the values on the data sheet.
- 3. Choose the appropriate size graduated cylinder (smallest size that will hold the object plus enough water). Add enough water to the graduated cylinder be able to cover your largest sample. Record the volume to the highest precision (0.1 mL or better).
- 4. Carefully add the sample to the graduated cylinder. There are two things to watch out for: breaking the cylinder and splashing water out. Tilting the cylinder and gently sliding the object in minimize both of these risks. Record the new volume.
- 5. Repeat with each sample piece. If the sample pieces are small, the pieces can remain in the graduated cylinder until all sample pieces have been added to the cylinder.
- 6. Determine the density of each piece and the average density.
- 7. Using Microsoft Excel<sup>®</sup>, graph the cumulative data. Use the largest values of mass and volume to determine your x and y scales.

#### Excel<sup>®</sup> Procedure

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

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



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

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

Name:			Section:	
Da	nta a	and Calculations for Experiment 2		
M	easu	rements		
A.	Те 1.	mperature Water at room temperature	°C	
	2.	Boiling point	°C	
	3.	Ice water Unstirred	°C	
		Stirred	°C	
	4.	Ice water with salt added	°C	
B.	Ма 1.	ass 100 mL beaker	g	
	2.	250 mL Erlenmeyer flask	g	
	3.	Weighing boat	g	
	4.	Mass of weighing boat + sodium chloride	g	
		Mass of sodium chloride (show calculation setup)	g	
C.	Le 1.	ngth Length of <i>←</i> →	cm	
	2.	Height of 250 mL beaker	cm	
	3.	Length of test tube	cm	
D.	Vo	olume		
	1.	200 mL mark (from Erlenmeyer flask) water transferred to graduated cylinder	mL	
	2.	Height of 5.0 mL of water in test tube	cm	
	3.	Height of 10.0 mL of water in test tube	cm	

#### Questions

- 1. Which would work better in this experiment as an unknown solid whose density is to be determined, wood chips or small quartz rocks? Explain your choice.
- 2. Why is it best to use a smaller graduated cylinder as opposed to a larger graduated cylinder for this experiment?
- 3. How well does the average density from the table and density from the slope of the graph compare? Which value is closer to the accepted density of your metal? (Refer to the *Handbook of Chemistry and Physics*). Calculate the percent error between your better value and the handbook value.

4. What is the density of a 9.343 gram piece of metal that causes the level of water in a graduated cylinder to rise from 5.1 to 8.1 mL when the metal is submerged in the water? Consider significant figures when doing the calculation.

# Experiment 3 – Graphing Freezing Points

#### Discussion

The freezing and melting of a substance occur at the same temperature, which is measured when the solid and liquid phases are at equilibrium (therefore, the terms "melting point" and "freezing point" can be used interchangeably). Accurate measurement of this temperature is useful for identification purposes, since few chemicals share exactly the same melting point. The unofficial definition of freezing point is the temperature where crystals first start to form. Officially, freezing point is determined by graphing a "cooling curve", which is a plot of temperature versus time as a substance is being cooled. The temperature will decrease at first as the liquid is cooled; then the temperature will remain constant during the phase change from liquid to solid. This "plateau" is the freezing point. When all the liquid has frozen, the temperature will again decrease.

An impurity will disrupt the crystal structure of a substance; therefore, it must be cooled below its normal freezing point to achieve crystallization. This depression of the freezing point has some interesting applications: measuring the amount of impurity, identification by mixing a known with an unknown substance, lowering the freezing point of water using antifreeze, etc.

Occasionally, a substance will become colder than its normal freezing point and still remain a liquid. This unstable condition is called "supercooling". "Shocking" the system by stirring or jarring usually will cause crystallization and the substance will rapidly rise to its normal freezing point.

The goal of this experiment is to determine freezing points by graphing two cooling curves: (1) pure (or glacial) acetic acid with stirring – this usually eliminates supercooling; and (2) pure acetic acid without stirring – supercooling will probably occur.

#### **Procedure**

#### **Trial 1: With Stirring**

- 1. Fasten a test tube clamp to a stand. Take a small-sized test tube and a stopper to the fume hood. Add 5 mL of acetic acid to the test tube and close the test tube with the stopper before removing from the fume hood.
- Place the test tube in the test tube clamp to minimize spillage. Adjust the temperature of the acetic acid to about 25 °C by warming or cooling the test tube in a beaker of water.



3. Prepare your cooling mixture by placing about 150 mL of ice in a 250 mL beaker; then add water to barely cover the ice.

- 4. Obtain the following equipment: Chemistry Department laptop (with Logger Pro software), Vernier LabQuest Mini, USB cable, thermometer probe. (*Note: an alternate procedure is available that uses LabQuest 2 devices instead of laptop computers.*)
- 5. Carefully insert the thermometer probe into the stopper & be sure the metal probe is touching the acetic acid.
- 6. Connect the thermometer probe to the LabQuest Mini (any CH) and then use the USB cable to connect the LabQuest Mini to the laptop.
- 7. Open the Logger Pro software.



3 Logger P	ro - Untitled			Data Callestian
ile Edit E	experiment Data Analyze	Insert Option	s Pa	Data Collection
No device	Start Collection Store Latest Run Clear Latest Run Keep Mark Data Tag Data	<space> Ctrl+L Ctrl+K d d</space>	0	Collection Triggering Mode: Time Based
2 3 4 5 6 7	Extend Collection (15 s) Connect Interface Remove Interface Set Up Sensors Add Offline Interface Data Collection.	Ctrl+T		Sampling Rate:         5       samples/minute       0.2       minutes/sample         Oversampling       Samples to be Collected: 51
8 9 10 11 12	Remote Change Units Calibrate Zero	Ctrl+0		· · ·
13 14	Live Readouts			Help Done Cancel

- 9. When you are ready to start the Trial, lower the test tube clamp until the acetic acid in the test tube is below the surface of the ice water and click the green Collect button.
- 10. Use the thermometer as a gentle stirrer during this trial. Continue recording temperatures for 10 minutes where Logger Pro will stop automatically. If the temperature has stabilized for several minutes (flat part of the graph) and the acetic acid is completely frozen, you may click the red Stop button to end the run.
- 11. To prepare the acetic acid for your next run, raise the test tube clamp and remove from the ice bath. Replace with a beaker of warm water to melt back to about 25 °C.
- 12. On the displayed graph, analyze the flat part. Press the mouse button and hold it down as you drag across the flat part to select it. Click Statistics. The mean temperature value for the selected data is listed in the statistics box on the graph. Record this value as the freezing temperature (in the Post Lab Questions). To remove the statistics box, click the upper-left corner of the box.

### **Trial 2: Without Stirring**

- 1. Prepare Logger Pro for your next trial: From the Experiment menu, choose Store Latest Run (to save for later comparison). You may either leave Trial 1 on the screen (Trial 2 data will overlay in another color of line) or you may hide the curve by: click the Temperature label of the graph, click More, and uncheck the Run 1 Temperature box. Click OK.
- 2. Replenish the ice bath if necessary.
- 3. Repeat the same timing procedure as Trial 1 but do not stir.
- 4. If the temperature falls to about 4 °C without forming crystals, you are observing supercooling. If supercooling persists for more than 2 minutes, "shock" the solution by moving the thermometer until crystals form.
- 5. Continue recording the temperature for the full 10 minutes or until completely frozen. When stopped, be sure to Store Latest Run.
- 6. On the displayed graph, analyze the flat part using the Statistics function again and record this value as the freezing temperature in the Post Lab.

#### **Completing the Graphs for your Experiment:**

- 1. If not already both displayed, click the Temperature axis label, click More, and check the Run 1 Temperature and Latest Temperature boxes. Click ok.
- 2. Label both curves by choosing Text Annotation from the Insert menu, and "Stirring" (or "Without Stirring") in the edit box. Then drag each box to a position near its respective curve. Adjust the orientation of the arrowhead by clicking and dragging to the desired position.
- 3. You will need to show both Trial 1 & Trial 2 graphs to your instructor by bringing the CHEM Dept. laptop to them for credit on this experiment.

### Hazardous Waste

Thaw the acetic acid with a beaker of warm water and then remove the temperature probe. Using your wash bottle filled with water, rinse the temperature probe off into a beaker from your drawer. Take the stoppered test tube, your wash bottle, and the beaker to the fume hood. Pour the acetic acid out of the test tube into the provided "Recycled Acetic Acid" bottle. Before you leave the fume hood, use your wash bottle to rinse the acetic acid out of the test tube into your beaker. *Please do not contaminate the recycled acetic acid with water!* You can then pour the water that is in your beaker down the sink drain and wash your test tube and beaker before you put them away.

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1.4	ame.

Section:	
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#### **Post-Lab Questions**

Us	Use your graphs to answer the following questions. Note: The accepted freezing point of acetic				
acid, according to the CRC Handbook of Chemistry and Physics, is:			16.6 °C		
1.	a.	What is the experimental freezing point of acetic acid in Trial 1?			

- b. What is the experimental freezing point of acetic acid in Trial 2?
- c. Calculate the percent error in the Trial 1 measurement of the freezing point:

SHOW CALCULATION:

2. What is supercooling? Did you observe supercooling in your experiment? Explain.

3. What is the difference between melting and freezing a substance?

## Experiment 4 – Hydrates and Anhydrous Salts

#### Discussion

When a salt incorporates a certain number of water molecules for each formula unit in order to create a stable complex substance, we have a hydrate. These compounds are not "wet"; the water molecules are incorporated into the solid structure in a fixed pattern. The following are some representative examples:

MgCl<sub>2</sub> 2H<sub>2</sub>O, Na<sub>2</sub>SO<sub>4</sub> 10H<sub>2</sub>O, CoCl<sub>2</sub> 6H<sub>2</sub>O, BaCl<sub>2</sub> 2H<sub>2</sub>O, CuSO<sub>4</sub> 5H<sub>2</sub>O

Notice that the formulas all begin with the salt and a dot separates the number of water molecules attached per formula unit. The water is chemically bound to the substance with weaker bonds than the ionic attraction within the salt or the covalent bonds within the water molecule.

In naming hydrates, you use the nomenclature rules you have learned to name the salt and add an appropriate prefix (mono = 1, di = 2, tri = 3, tetra = 4, penta = 5, hexa = 6...) to identify the number of water molecules ending the name with the word hydrate. For example,  $CuSO_4 \cdot 5H_2O$  has the name copper(II) sulfate pentahydrate.

The water chemically bound in hydrates can generally be removed by heating without destroying the salt or the molecule of water. When the water has been completely removed, we call the remaining compound the anhydrous salt (*salt without water*). Anhydrous salts will readily absorb water from its surroundings and may be used as moisture detectors. A common example is anhydrous cobalt(II) chloride, CoCl<sub>2</sub> (blue in color). When in contact with moisture, this substance absorbs water to create cobalt(II) chloride hexahydrate, CoCl<sub>2</sub> 6H<sub>2</sub>O (pink in color).

In this experiment, hydrated salts will be heated. First, we will perform a qualitative test with the hydrate  $CuSO_4$  5H<sub>2</sub>O (blue in color), whereby heat is applied until the hydrate becomes anhydrous CuSO<sub>4</sub> (white in color). Next, the resulting liquid will be tested with anhydrous CoCl<sub>2</sub> to detect if that liquid is water. Finally, we will also perform a quantitative experiment with an unknown hydrate to determine the mass percent of water lost and solve for the formula and name of the unknown hydrate.

#### **Procedure**

- A. Qualitative Determination of the Released Liquid after Heating CuSO4<sup>-5</sup>H<sub>2</sub>O
  - 1. Weigh out approximately 3 grams of copper(II) sulfate pentahydrate into a clean, dry large ignition tube (25 x 200 mm). Make sure the hydrate is not sticking to the sides; it should all be near the bottom of the tube.
  - 2. Clamp the ignition tube at a 15–20 degree downward from the horizon so that the mouth of the tube is lower than the bottom of the tube where the hydrate is located. The clamp should be near the mouth of the test tube.

- 3. To collect a sample of the liquid that will be removed from the hydrate, place a medium test tube held upright in an Erlenmeyer flask just below the mouth of the ignition tube containing the hydrate. Your instructor will demonstrate this set-up.
- 4. Heat the hydrate with a Bunsen burner gently at first, and gradually increase the intensity of the heat. Note the changes that occur. Collect some liquid that condenses in the lower, cooler mouth of the tube. Continue heating until most of the blue color has disappeared. Your residue should now be pale blue or white (*not black, see note*). To remove the remaining liquid still inside the ignition tube, gently warm the entire tube to vaporize. Allow tube and contents time to cool. Observe and record the appearance and odor of the collected liquid. <u>NOTE</u>: at temperatures above 600 °C, the copper(II) sulfate decomposes; sulfur trioxide is driven off and black copper(II) oxide remains as residue.
- 5. Obtain a couple pieces of anhydrous cobalt(II) chloride test strips. Place distilled water on one and your experiment liquid on the other. Compare and record observations.
- 6. Empty the anhydrous salt from your cooled ignition tube onto a watch glass and divide it into two sections. To one section, add a few drops of distilled water; to the other, add your experiment liquid. Compare and record observations. Dispose of residue in the waste container provided.
- B. Quantitative Determination of Mass Lost in a Hydrate
  - 1. Obtain an unknown hydrate sample. Record the unknown number.
  - 2. Weigh a clean dry crucible with its cover to the highest precision. Be sure that you use the same balance for all mass measurements within the same experiment to cancel out systematic errors.
  - 3. Add between 2–3 grams of the unknown into the crucible with cover and weigh to the highest precision.
  - 4. To a ring stand, attach a ring and place a clay triangle on the ring. Transfer the crucible onto the clay triangle and adjust the cover so it is slightly ajar to allow liquid vapor to escape. Very gently heat with a Bunsen burner for about 5 minutes. Readjust the flame to continue heating, more intensely now so that the bottom of the crucible glows red, for another 12 minutes.
  - 5. After this two-step heating process (1<sup>st</sup> heating) is complete, turn off the burner, close the crucible lid, and allow the crucible to cool for about 10 minutes before weighing. It is important to have all mass measurements made at or near room temperature. Do not transfer the hot crucible to the tabletop; it is too hot and will permanently mark the table. Use the crucible cooling plates that are provided. Weigh your sample.
  - 6. Heat the covered crucible and contents another 6 minutes at maximum temperature; cool and reweigh (2<sup>nd</sup> heating). If the results agree within  $\pm 0.050$  g, you are done and will not need to have a 3<sup>rd</sup> heating. If the difference is greater than  $\pm 0.050$  g, repeat the heating for another 6 minutes (3<sup>rd</sup> heating).

#### **Data and Calculations for Experiment 4**

- A. Qualitative Determination of the Released Liquid
  - 1. Record observations regarding the solid before, during, and after heating the copper(II) sulfate pentahydrate.
  - 2. Compare and record observations after adding liquid to the anhydrous cobalt(II) chloride test strips.
  - 3. Compare and record observations after adding liquid to the residue on the watch glass.
  - 4. What conclusions can you draw from the above observations?
  - 5. Write the balanced chemical equation for the decomposition of copper(II) sulfate pentahydrate, include phases.
- B. Quantitative Determination of Mass Lost in a Hydrate

	Sample number:	
1.	Mass of crucible and cover	
2.	Mass of crucible, cover and sample	
3.	Mass of crucible, cover and sample after 1 <sup>st</sup> heating	
4.	Mass of crucible, cover and sample after 2 <sup>nd</sup> heating	
5.	Mass of crucible, cover and sample after 3 <sup>rd</sup> heating	
6.	Mass of sample after final heating	
7.	Mass of original sample	
8.	Total mass lost by sample	
9.	Percentage of water in sample	

10. Ask your instructor for the name of the anhydrous salt of your residue and solve for the formula and name of your original unknown hydrate.

Formula: \_\_\_\_\_\_ Name: \_\_\_\_\_

11. Is it possible that the decrease in mass from heating is something other than water? *Yes* or *No* Explain and include an example.

**<u>Pre-Lab</u>** (to be completed before coming to lab)

A student heated a hydrated salt sample with an initial mass of 2.244 grams. After the second heating, the mass had decreased to 1.798 grams. Make the assumption that all the lost mass is water.

- a) Solve for the mass lost.
- b) Calculate the percentage of water in the original hydrated salt sample.
- c) The instructor has informed you that the sample is a hydrate of ZnSO<sub>3</sub>. Use the information above and molar masses from the periodic table to solve for the moles of ZnSO<sub>3</sub> in the residue and the moles of H<sub>2</sub>O lost.
- d) Solve for the *X* in the formula of the hydrate of  $ZnSO_3 XH_2O$  and name it. Moles of water / moles of  $ZnSO_3 = X$

Formula: \_\_\_\_\_

Name: \_\_\_\_\_

Chemistry M12 Laboratory Manual

# Experiment 5 – Properties of Solutions

#### **Discussion**

In today's lab, you'll investigate the qualitative nature of solutions. The first step is learning some common terms.

**Solute** refers to a compound that **dissolves** in a solvent to form a solution. A **solution** can have one or more solutes, but only one solvent. The **solvent** is the compound that is predominant in the solution. A solute is said to be dissolved when it forms a clear, but not necessarily colorless, liquid. Thus, sugar dissolves in water, but fine sand and dust form **suspensions** which are not true solutions.

Solvents can be sorted by their **polarity**. Water is very polar, while benzene, decane, and gasoline are considered **non-polar**. The term organic solvent refers to most solvents other than water that are carbon-containing. Organic solvents can be either polar or non-polar, depending upon their structure. For example, methanol and ethanol are polar organic solvents, while ether and acetone are less polar, and decane and benzene are considered non-polar organic solvents.

Solubility is a measure of how much of a compound can eventually dissolve in a solvent. If a solid does not dissolve, the compound is said to be **insoluble**. It can also be described as slightly soluble, moderately soluble, or very soluble. If the compound is a liquid (not a solid) it can dissolve and is described as **miscible**, or instead forms two layers and is called **immiscible**. Ethanol and water are miscible, while oil and water are immiscible.

Concentration refers to the amount of solute relative to the total volume of solution. A dilute solution has little solute per 100 grams of solution, while a concentrated solution has more solute. A solution is considered saturated when no more solute can dissolve in that solution without it precipitating thereafter.

A supersaturated solution is a solution that holds more solute than it normally can hold at that temperature. Given time, some solute will precipitate out of solution. In other words, the solution is unstable over time.

Concentration can be measured using several terms. "Proof" is used to measure alcohol content in liquor and beer. Chemists tend to use mass percent and molarity which are defined below. Remember that mass percentages range from 0 to 100%, and molarities are generally less than 18 M. Very few compounds can form solutions with higher concentrations.

Mass Percent of  $\mathbf{X} = \frac{\text{mass of } \mathbf{X}}{\text{mass of } \mathbf{X} + \text{mass of solvent}} \times 100 \%$ 

#### **Procedure**

A. Concentration of a Saturated Solution.

In this section, you'll figure out how many grams of potassium chloride per mL of solution were present in a pre-made saturated solution of KCl.

- 1. Weigh a clean, dry evaporating dish. In this dish, add 6.0 mL of solution and weigh again. Place the dish in a 250-mL beaker of boiling water. Evaporate the solution until a white solid is present in the dish. Don't let the boiling water bath go dry. This step will take approximately half an hour.
- 2. Remove the dish from the boiling water with tongs. Place the dish on a wire mesh and gently heat with a Bunsen burner. If you heat too strongly, the solid may "pop" and you will lose some.
- 3. Let the dish cool until it can be touched safely. Weigh the dish to find out how many grams of potassium chloride are present.

Cleanup: Wash the solid down the drain.

B. Relative Solubility of a Solute

In this section, you will determine whether iodine, a reddish solid, dissolves better in water or decane.

- 1. Take a test tube and add about 5 mL of water and 2 mL of decane. Stopper the test tube and give it a gentle shake. Note which layer was on top.
- 2. To this tube, add 5 mL of saturated iodine-water solution. Gently shake again and see which layer has more color.

Cleanup: Empty the test tube into the waste labeled "Decane Waste".

C. Miscibility of Liquids

In this section, you will find out what liquids are miscible with water.

Take three dry test tubes and add the following pair of liquids. Stopper the test tubes and gently shake them. Are there two layers or one?

- 1. 1 mL of kerosene and 1 mL of isopropyl alcohol
- 2. 1 mL of kerosene and 1 mL of water
- 3. 1 mL of isopropyl alcohol and 1 mL of water

Dispose of the first two kerosene mixtures in the "Kerosene Waste" container.

- D. Effect of Particle Size on Rate of Dissolution
  - 1. Fill a test tube with about 0.5 cm of fine crystals of sodium chloride. Fill a second test tube with about 0.5 cm of coarse crystals of sodium chloride. Add 10 mL of water to each tube and shake both tubes an equal number of times. Shake both tubes equally. Time how long it takes to dissolve each.

These solutions can be disposed of down the sink.

- E. Effect of Temperature on Dissolution
  - 1. Weigh out two 0.5 g samples of fine sodium chloride crystals. Take two 250-mL beakers and add 50 mL of water to them. Heat one of the beakers to boiling, then let it cool for one minute.
  - 2. Add the salt samples to each beaker and time how long it takes to dissolve each.
  - 3. As soon as the salt dissolves, gently swirl the hot water and observe the denser salt layer in the bottom of the flask. Repeat the process with the cold water.

These solutions can be disposed of down the sink.

- F. Solubility versus Temperature; Saturated and Unsaturated Solutions
  - 1. Weigh out 1.0 g of NaCl and 1.0 g of NH<sub>4</sub>Cl and place them in separate, labeled test tubes and add 5 mL of water. Stopper the test tubes and shake the tubes until the salts dissolve.
  - 2. Add another 1.4 g of NaCl to the NaCl solution, and another 1.4 g of NH<sub>4</sub>Cl to the NH<sub>4</sub>Cl solution. Stopper and shake the tubes for 3 minutes. Note whether or not the salts dissolved.
  - 3. Remove the stoppers and place both tubes in a beaker of boiling water, gently shaking occasionally, and note the results after 5 minutes.
  - 4. Remove the tubes and cool with running tap water for one minute and record your observations. Let the solutions stand for a few minutes and record your observations.

Pour the solutions down the drain.

- G. Ionic Reactions in Solution
  - 1. Place a small lump of pea-sized quantities of a) barium chloride, b) sodium sulfate, c) sodium chloride, and d) barium sulfate into four separate labeled test tubes.
  - 2. Add 5 mL of water, stopper the tubes, and shake them. Which sample(s) do(es) not dissolve?
  - 3. Mix the barium chloride and sodium sulfate together and note the results.
  - 4. Write an equation that describes the results of these test tubes being mixed.

Dispose of all solutions in the "Barium waste" container.

## Data and Calculations for Experiment 5

A. Concentration of a Saturated Solution (record all masses as x.xxx g)

	1.	a)	Mass of evaporating dish	
		b)	Mass of evap. dish and potassium chloride solution	
		c)	Mass of evap. dish and residue	
	2.	Ca a)	lculate: (show setups) Mass of potassium chloride solution	
		b)	Mass of residue	
		c)	Mass of water in potassium chloride solution	
		d)	Mass percent of potassium chloride in the solution	
		e)	Grams of potassium chloride per 100 g of water in the solution	
B.	Re	lativ	ve Solubility of a Solute in Two Solvents	
	1.	a)	Which liquid is denser, decane or water?	
		b)	How did you decide which layer was water?	
	2.	WI	hat is the color of iodine in water?	
		W	hat is the color of iodine in decane?	
	3.	W	nich solvent dissolves more iodine? How did you decide this?	

- C. Miscibility of Liquids
  - 1. Which liquids were miscible with each other?
  - 2. Which liquids were immiscible with each other?
- D. Particle Size and Dissolution Rates
  - 1. How long did it take the fine salt crystals to dissolve?
  - 2. How long did it take the coarse salt crystals to dissolve?
  - 3. Based on these observations, how does particle size affect the rate at which a substance is able to dissolve?
- E. Temperature and Dissolution Rates
  - 1. How long did it take the salt crystals to dissolve in hot water?
  - 2. How long did it take the salt crystals to dissolve in cold water?
  - 3. Based on these observations, how does temperature affect the rate at which a substance is able to dissolve?
- F. Temperature and Solubility
  - 1. Was the solution with 1.0 g of NaCl in 5.0 mL water saturated at room temperature?
  - 2. Was the solution with 1.0 g of NH<sub>4</sub>Cl in 5.0 mL water saturated at room temperature?
  - 3. Was the solution with 2.4 g of NaCl in 5.0 mL water saturated at room temperature?
  - 4. Was the solution with 2.4 g of NH<sub>4</sub>Cl in 5.0 mL water saturated at room temperature?

- 5. Which salt was least soluble at higher temperatures?
- 6. At the higher temperatures, was the NaCl solution saturated?
- 7. At the higher temperatures, was the NH<sub>4</sub>Cl solution saturated?
- 8. What happened to the NaCl solution when it was cooled back to room temperature?
- 9. What happened to the NH<sub>4</sub>Cl solution when it was cooled back to room temperature?
- 10. Solubility is defined as the amount of solute that can dissolve in a given quantity of solvent. Based on your observations in this part, how does temperature affect the solubility of solid solutes? Does it affect different substances in identical ways?

- G. Ionic Reactions in Solution
  - 1. Write the formulas for the following substances. Include states of matter (e.g. (aq) or (s)) based on the results of your experiment:

barium sulfate	
barium chloride	

sodium sulfate

sodium chloride

- 2. Write the equation that shows the reaction of barium chloride and sodium sulfate. Use state indicators (e.g. (aq) or (s)) for all compounds.
- 3. Which compound is the white precipitate? How do you know this?

## Experiment 6 – Decomposition of Potassium Chlorate

#### **Discussion**

The percentage composition of a compound is the percent by mass of each element in the compound. This can be theoretically calculated using atomic masses found on the periodic table, and experimentally calculated from lab data. The percent composition is calculated by taking the mass of the part divided by the mass of the whole times 100.

In this experiment, solid potassium chlorate will be strongly heated. This causes it to decompose into solid potassium chloride and oxygen gas. The relative amount of potassium chloride (residue left behind) and oxygen gas (mass lost) are experimentally measured and compared to the theoretical values.

Percent error is calculated by finding the absolute value of the difference between the theoretical and experimental values and dividing that number by the "true" theoretical value and last multiplying the answer by 100.

Following the quantitative test, we will perform a qualitative test on the residue and known compounds of  $KClO_3$  and KCl using silver nitrate solution. AgNO\_3 solution is used as a general test for chloride ions. A positive test forms white solid AgCl and indicates the presence of Cl<sup>-</sup> ions in a solution. Tap water contains chloride ions, so it is very important to perform this test with clean equipment that has been rinsed out with deionized water and to use deionized water in the test.

To obtain accurate and precise data you should do the following:

- 1. Use the same balance for all mass measurements.
- 2. Record data directly and immediately into the lab report.
- 3. Record data completely (to the nearest 0.001 g); do not round off the data.
- 4. If an error is made in data collection, do not scratch out or erase but simply draw a single line through the error and write the correction beside it.
- 5. Duplicate samples should be analyzed so that results can be compared and averaged.
- 6. Handle the crucibles with tongs not fingers.
- 7. The crucible must be covered while heating to avoid any loss due to splatter.

## **Procedure**

- A. Quantitative Determination of Percent Composition
  - 1. Weigh a clean dry crucible and cover to the highest precision. Be sure that you use the same balance for all mass measurements within the same experiment to cancel out systematic errors.
  - 2. Carefully read the label on the container and add between 1.0 to 1.5 grams of potassium chlorate, KClO<sub>3</sub>, into the crucible and cover and weigh to the highest precision.

<u>NOTE</u>: In part B of this lab, we will be using some potassium chloride. If you accidentally place KCl in your crucible, no mass will be lost on heating and you will have to start over and repeat the experiment correctly using  $KClO_3$ !

- 3. To a ring stand, attach a ring and place a clay triangle on the ring. Place the crucible with cover on the clay. Very gently heat with a Bunsen burner for about 8 minutes. Readjust the flame and continue heating more intensely now so that the bottom of the crucible glows red for another 10 minutes.
- 4. After this two step heating process (1<sup>st</sup> heating) is complete, turn off the burner, close the crucible lid, and allow to cool about 10 minutes before weighing. It is important to have all mass measurements made at or near room temperature. Do not transfer the hot crucible to the tabletop; it is too hot and will permanently mark the table. Use the crucible cooling plates that are provided. Weigh your sample. The cooling period is a great time to start the second sample.
- 5. Heat the covered crucible and contents another 6 minutes at maximum temperature, then cool and reweigh ( $2^{nd}$  heating). If the results agree within  $\pm 0.050$  g, you are done and will not need to have a  $3^{rd}$  heating. If the difference is greater than 0.050 g, repeat the heating for another 6 minutes ( $3^{rd}$  heating).
- B. Qualitative Examination of the Residue
  - 1. Place three clean test tubes in a rack. Put a pea sized quantity of KCl in test tube number 1 and a pea sized quantity of KClO<sub>3</sub> in the test tube number 2. Add 10 ml of deionized water to each and mix. Next, add 10 ml of deionized water to your residue in the crucible, mix, and transfer a portion to test tube number 3.

<u>NOTE</u>: It is not necessary to dissolve and transfer the entire residue.

2. Add 3 drops of 6 M HNO<sub>3</sub> and 5 drops of 0.1 M AgNO<sub>3</sub> solution to each test tube. Record observations.

DISPOSE of solutions and precipitates containing silver in the waste container provided.

#### Section:

#### Data and Calculations for Experiment 6

- A. Quantitative Determination of Percent Composition
  - 1. When solid KClO<sub>3</sub> is heated above 400  $^{\circ}$ C, it decomposes to solid potassium chloride and elemental oxygen gas. Write the balanced equation for the decomposition of KClO<sub>3</sub> solid.
  - 2. What is the remaining residue in the crucible after heating?
  - 3. What substance is lost during the heating?

		Sample 1	Sample 2
4.	Mass of crucible and cover		
5.	Mass of crucible, cover and sample		
6.	Mass of crucible, cover and sample after 1 <sup>st</sup> heating		
7.	Mass of crucible, cover and sample after 2 <sup>nd</sup> heating		
8.	Mass of crucible, cover and sample after 3 <sup>rd</sup> heating		
9.	Mass of original sample		
10.	Mass of the residue		
11.	Mass lost upon heating		
12	Experimental percentage of KCl in the KClO <sub>3</sub> sample	2.	
	Sample 1:		

Sample 2:

13. Experimental percentage of oxygen in the KClO<sub>3</sub> sample.Sample 1:

Sample 2:

14. Using the atomic masses from the periodic table, solve for the molar mass of KClO<sub>3</sub>.

15. Theoretical percentage of KCl in the KClO<sub>3</sub> sample

16. Theoretical percentage of oxygen in the KClO<sub>3</sub> sample

17. Percent error in oxygen determination Sample 1:

Sample 2:

B. Qualitative Examination of the Residue

1. Record what you observed when AgNO<sub>3</sub> solution was added to the following:

i. KCl

ii. KClO<sub>3</sub>

iii. Residue

2. What does the evidence lead you to believe about the residue?

3. Does the evidence from the AgNO<sub>3</sub> test prove conclusively (without a doubt) that the residue is KCl? Explain.

Chemistry M12 Laboratory Manual

**<u>Pre-Lab Assignment</u>** (to be completed before coming to lab)

- 1. a) Write the balanced equation for the decomposition of  $Mg(ClO_3)_2$  solid.
  - b) A student heated 1.228 grams of Mg(ClO<sub>3</sub>)<sub>2</sub> until a stable weight was determined. The remaining residue weighed 0.584 grams. Solve for the experimental percentage of oxygen.
  - c) Calculate the theoretical percentage of oxygen in Mg(ClO<sub>3</sub>)<sub>2</sub>.
  - d) Calculate the percent error in oxygen determination.
- 2. Given the mass percent of each element:

18.8% Na 29.0% Cl 52.2% O

Solve for the empirical formula and name it.

- 3. Predict the products and balance the equations for the following decomposition reactions:
  - a) NaClO<sub>3</sub>(s)  $\rightarrow$
  - b)  $Ca(ClO)_2(s) \rightarrow$
  - c)  $Al(ClO_3)_3(s) \rightarrow$
  - d)  $Mg(ClO_2)_2(s) \rightarrow$

# Experiment 7 – Double Displacement Reactions

#### **Discussion**

In this experiment, double displacement reactions will be studied, where two water solutions, each containing positive and negative ions, will be combined. Consider the generalized reaction shown below:

$$AB + CD \rightarrow AD + CB$$

where AB exists as  $A^+$  and  $B^-$  ions in solution, and CD exists as  $C^+$  and  $D^-$  ions in solution. Each of the positive ions can combine with the negative ion of the other compound as shown above. But the question then becomes: has there been a reaction? To answer this question, we look at the products. Is either one an insoluble compound giving a precipitate (information available from a solubility table), is either one a gas or producer of a gas, or would a temperature change be predicted? Has a weak electrolyte such as a weak acid been formed? If no to all of these, then no reaction occurs; this is simply a mixture. If yes to any one or more of these, then a reaction occurs.

#### **Procedure**

Each part of the experiment below consists of mixing equal volumes of two solutions from dropper bottles in a 24 well-plate. Place 5 drops of each indicated chemical in the well-plate. Write your observations on the report sheet. Note the formation of any precipitate or gas. If neither results, test the well-plate with a thermometer for any temperature change. If no change is noted, write NR (No Reaction) for the mixture.

- 1. Mix 5 drops of 0.1 M NaCl(aq) with 5 drops of 0.1 M KNO<sub>3</sub>(aq).
- 2. Mix 5 drops of 0.1 M NaCl(aq) with 5 drops of 0.1 M AgNO<sub>3</sub>(aq).
- 3. Mix 5 drops of 0.1 M Na<sub>2</sub>CO<sub>3</sub>(aq) with 5 drops of dilute 6 M HCl(aq).
- 4. Mix 5 drops of 10% NaOH(aq) with 5 drops of dilute 6 M HCl(aq).
- 5. Mix 5 drops of 0.1 M BaCl<sub>2</sub>(aq) with 5 drops of dilute 3 M H<sub>2</sub>SO<sub>4</sub>(aq).
- 6. Mix 5 drops of dilute 6 M  $NH_4OH(aq)$  with 5 drops of dilute 3 M  $H_2SO_4(aq)$ .
- 7. Mix 5 drops of 0.1 M CuSO<sub>4</sub>(aq) with 5 drops of 0.1 M Zn(NO<sub>3</sub>)<sub>2</sub>(aq).
- 8. Mix 5 drops of 0.1 M Na<sub>2</sub>CO<sub>3</sub>(aq) with 5 drops of 0.1 M CaCl<sub>2</sub>(aq).
- 9. Mix 5 drops of 0.1 M CuSO<sub>4</sub>(aq) with 5 drops of 0.1 M NH<sub>4</sub>Cl(aq).
- 10. Mix 5 drops of 10% NaOH(aq) with 5 drops of dilute 6 M HNO<sub>3</sub>(aq).
- 11. Mix 5 drops of 0.1 M FeCl<sub>3</sub>(aq) with 5 drops of dilute 6 M NH<sub>4</sub>OH(aq).
- 12. IN THE HOOD: Add a small amount of Na<sub>2</sub>SO<sub>3</sub>(s) to 5 drops of water and mix to dissolve. Add 5 drops of dilute 6 M HCl(aq) to the first solution.

Dispose of all solutions in the appropriate WASTE CONTAINER in the hood.

Section:

#### Data for Experiment 7

Record your observations for each combination below. If a reaction occurs, write balanced MOLECULAR, IONIC, and NET-IONIC equations. If no reaction occurs, write NR. Make sure to include the physical states of all the products.

1. NaCl(aq) and KNO<sub>3</sub>(aq)

Observations:

Molecular:

Ionic:

Net-Ionic:

2. NaCl(aq) and AgNO<sub>3</sub>(aq)

Observations:

Molecular:

Ionic:

Net-Ionic:

3. Na<sub>2</sub>CO<sub>3</sub>(aq) and HCl(aq)

Observations:

Molecular:

Ionic:

Net-Ionic:

4. NaOH(aq) and HCl(aq)

Observations:

Molecular:

Ionic:

#### Name: \_\_\_\_\_

Section: \_\_\_\_\_

5.  $BaCl_2(aq)$  and  $H_2SO_4(aq)$ 

**Observations**:

Molecular:

Ionic:

Net-Ionic:

6.  $NH_4OH(aq)$  and  $H_2SO_4(aq)$ 

Observations:

Molecular:

Ionic:

Net-Ionic:

7.  $CuSO_4(aq)$  and  $Zn(NO_3)_2(aq)$ 

Observations:

Molecular:

Ionic:

Net-Ionic:

8. Na<sub>2</sub>CO<sub>3</sub>(aq) and CaCl<sub>2</sub>(aq)

**Observations**:

Molecular:

Ionic:

#### Name: \_\_\_\_\_

Section: \_\_\_\_\_

9.  $CuSO_4(aq)$  and  $NH_4Cl(aq)$ 

**Observations**:

Molecular:

Ionic:

Net-Ionic:

## 10. NaOH(aq) and HNO<sub>3</sub>(aq)

Observations:

Molecular:

Ionic:

Net-Ionic:

### 11. FeCl<sub>3</sub>(aq) and NH<sub>4</sub>OH(aq)

Observations:

Molecular:

Ionic:

Net-Ionic:

12. Na<sub>2</sub>SO<sub>3</sub>(aq) and HCl(aq)

**Observations**:

Molecular:

Ionic:

#### **Questions**

- 1. For each of the reactions listed below, write balanced molecular, ionic, and net-ionic equations. If no reaction occurs, write NR. Assume all reactants are aqueous unless otherwise noted. Include all physical states.
  - A. Lead(II) nitrate and magnesium sulfate solutions are combined.

Molecular:

Ionic:

Net-Ionic:

B. Barium chloride solution is poured into a solution of ammonium carbonate.

Molecular:

Ionic:

Net-Ionic:

C. Magnesium chloride solution is mixed with nickel(II) nitrate solution.

Molecular:

Ionic:

Net-Ionic:

D. Cobalt(II) sulfate and lithium sulfide solutions are combined.

Molecular:

Ionic:

Net-Ionic:

E. Hydrochloric acid solution is reacted with a solution of lithium carbonate.

Molecular:

Ionic:

F. Hydroiodic acid and ammonium sulfite solutions are mixed.

Molecular:

Ionic:

Net-Ionic:

G. Sodium hydroxide solution is poured into a solution of cobalt(II) chloride.

Molecular:

Ionic:

Net-Ionic:

H. Ammonium chloride and potassium hydroxide solutions are reacted.

Molecular:

Ionic:

Net-Ionic:

I. Solid strontium bromide is mixed with a solution of potassium phosphate.

Molecular:

Ionic:

Net-Ionic:

J. Solutions of ammonium sulfate and sodium chloride are combined.

Molecular:

Ionic:

# Experiment 9 – Ionization and the Nature of Acids, Bases, and Salts

#### Discussion

Compounds were defined by Sven Arrhenius to be acids if they release  $H^+$  ions in solution when dissolved. This modern definition replaced older definitions based on taste (i.e acids tend to be sour tasting) or if they changed litmus paper's color. Bases (which tend to taste bitter) were defined as compounds that give up  $OH^-$  (hydroxide) ions in water. This definition was limited to compounds in water and gives way to Brønsted-Lowry acid-base theory.

Brønsted-Lowry acid-base theory keeps the definition of an acid as something that donates an  $H^+$  ion and defines bases as anything that accepts the  $H^+$  ion. Acids become proton donors; bases become proton acceptors. In any acid-base equation, there will be one acid and one base *on each side* of the equation. Which compound is an acid depends on whether that compound is donating or accepting a proton.

 $NH_3 + H_2O \rightarrow NH_4^+ + OH^-$ Base Acid Conj. Acid Conj. Base  $HCl + H_2O \rightarrow Cl^- + H_3O^+$ Acid Base Conj. Base Conj. Acid

Water can function as both an acid and a base, depending on the other reagents!

HCl(aq)	Hydrochloric acid	$H_2SO_4$	Sulfuric acid
HBr(aq)	Hydrobromic acid	$HC_2H_3O_2$	Acetic acid
HI(aq)	Hydroiodic acid	H <sub>2</sub> CO <sub>3</sub>	Carbonic acid
H <sub>3</sub> PO <sub>4</sub>	Phosphoric acid	HNO <sub>3</sub>	Nitric acid

Many common strong bases contain hydroxides (OH<sup>-</sup>) and a metal.

Sodium hydroxide
Potassium hydroxide
Calcium hydroxide
Magnesium hydroxide
Ammonium hydroxide (best written as NH <sub>3</sub> ·H <sub>2</sub> O)

Solutions that contain bases are called *alkali* or *alkaline*, from an Arabic word for "ashes". Campfire ashes ("bitter ashes") contain hydroxides and carbonates of potassium and sodium, which form basic or alkaline solutions. Compounds from plants that dissolve in water to form alkaline solutions are called *alkaloids*. A common example of a bitter-tasting alkaloid is caffeine.
Name: \_

Section:

The term pH is used to measure the concentration of an acid in water. Thus, it is important to remember that one acid can produce a range of pH values, depending upon the amount of acid relative to the volume of solution. pH is defined by the equation  $pH = -log [H^+]$ . Therefore, a solution of 1.0 M HCl will produce 1.0 M H<sup>+</sup> ions, assuming the HCl breaks up entirely. Since log [1.0] = 0, the pH of this solution is 0. The pH of pure water will be 7.0, while the pH of a very basic solution can be above 14.

рŀ	I < 7 acidic solutions	pH = 7 neutral solution	pH > 7 basic solution
P *		pri , neudul solution	

When acids react with bases, the  $H^+$  from the acid and the  $OH^-$  from the bases "cancel" each other and form water molecules ("HOH"). The anions of the acid and the cations from the base combine to form ionic compounds or salts. For example, consider the reaction of sulfuric acid with sodium hydroxide:

$$H_2SO_4(aq) + 2NaOH(aq) \rightarrow Na_2SO_4(aq) + 2H_2O(l)$$

## **Reactions of Oxides with Water**

The oxides of elements often react with water to form new compounds. Depending upon which family the element is in, the new compound may be acidic or basic. For example, sulfur can be oxidized to form sulfur trioxide, which reacts with water to make sulfuric acid. Consider the following balanced equations:

$$S + O_2 \rightarrow SO_2$$
  
 $2SO_2 + O_2 \rightarrow 2SO_3$   
 $SO_3 + H_2O \rightarrow H_2SO_4$ 

Carbon dioxide reacts with water to form carbonic acid as follows:

$$CO_2 + H_2O \rightarrow H_2CO_3$$

The metal oxides react with water to form basic compounds. Calcium oxide reacts with water to form calcium hydroxide, while magnesium oxide reacts with water to form magnesium hydroxide:

$$CaO + H_2O \rightarrow Ca(OH)_2$$
  
MgO + H\_2O  $\rightarrow$  Mg(OH)\_2

Name: \_\_\_\_

### Electrolytes

Surprisingly, pure distilled water does not conduct electricity. In order for a charge to pass through water, it needs to be carried by positive and negative charges. The more charges, the more current can pass. If the charges cannot move, as in solid salts with no water present, then electricity cannot be conducted.

Compounds can be divided into strong electrolytes, weak electrolytes, and non-electrolytes depending upon how well they conduct electricity when dissolved in solution. Remember that compounds that don't dissolve in the solvent shouldn't be called electrolytes at all. For example, iron bars, wood, or plastics are not electrolytes regardless of whether they conduct electricity or not.

In a strong electrolyte, the compound breaks up into cations or anions in a process called "dissociation". In a weak electrolyte, some of the compound dissociates into ions, even though the entire compound dissolves. In non-electrolytes, the compound dissolves but does not break up at all.

#### **Procedure**

A. Electrolytes

In this part of the experiment, your instructor will demonstrate the conductivity of various solutions and reactions.

- B. Investigating Acids
  - 1. Reactions of Acids with Metals
    - a. Take four separate test tubes and place 5 mL of 6 M HCl in tube #1, 3 M H<sub>2</sub>SO<sub>4</sub> in tube #2, 6 M HNO<sub>3</sub> in tube #3, and 6 M acetic acid in tube #4.
    - b. Put roughly a 2 cm strip of magnesium metal into each tube. Record the results.
    - c. As the metal is still bubbling, place a glowing piece of wood (splint) into the test tube.
  - 2. Measurement of pH and Acidity
    - a. Place 5 mL of water in a test tube and add 2 drops of a phenolphthalein indicator solution in it. Add a few drops of dilute hydrochloric acid and record what happens.
    - b. There are three solutions of HCl prepared in front of the classroom. The most concentrated, 0.1 M HCl, is one hundred times more concentrated than the weakest solution, the 0.001 M HCl. Use the pH meter to record the pH's of the three solutions.

- 3. Reactions of Acids with Carbonates and Bicarbonates
  - a. Take a 100 mL beaker and just cover the bottom with a thin layer of sodium bicarbonate (baking soda). Add about 4 to 5 mL of diluted (6 M) HCl to the beaker. Record the results. Lower a lit match into the beaker and record what happens.
  - b. Try the above reaction again with a chip of calcium carbonate (limestone, marble). Let the reaction go for about 2 minutes before lowering a lit match into it. When completed, throw them in the labeled water container; DO NOT CLOG THE DRAIN!
- 4. Neutralizing Acids with Base: Using Indicators

In this experiment, you will make water acidic and then basic to see how the pH affects a common indicator solution.

Add 25 mL of water and 3 drops of a phenolphthalein solution to a 100 mL beaker, and then add 5 drops of 6 M hydrochloric acid. To this solution, add 10 percent sodium hydroxide solution drop by drop until the indicator changes color. Once you've gotten this color change, reverse it by adding more dilute acid dropwise.

5. Reaction of a Non-Metal Oxide and Water

In this section, you'll investigate what happens when an oxide of a non-metal, sulfur, reacts with water.

a. This part of the experiment must be done in the fume hood! Place a small lump of sulfur in a deflagrating spoon (which looks like a ladle with a long handle) and set it on fire with a Bunsen burner. Once the sulfur is burning, lower the spoon into a bottle containing 15 mL of water; this will allow the fumes of combustion to fill the air space of the bottle. After 2 minutes, remove the sulfur and cover the bottle with a glass plate. Shake the bottle to mix the gas and water. Is the water acidic or basic?



b. In a test tube, generate carbon dioxide gas by treating marble chips with hydrochloric acid (see section 3b). Bubble the gas into another beaker containing 10 mL of water, 2 drops of 10% sodium hydroxide, and a few drops of phenolphthalein indicator.



- C. Properties of Bases and Basic Solutions
  - 1. Properties of ammonium and sodium hydroxides
    - a. Place three drops of concentrated ammonium hydroxide (used in "Windex" cleaners) in 10 mL of water in a test tube. In another test tube, place three drops of concentrated sodium hydroxide (used in "Drano" pipe cleaners) in 10 mL of water. Rub a few drops of the diluted solution from each test tube onto your fingers. What is the difference in feeling between the two solutions? Wash your hands with water afterwards until your skin feels normal.
    - b. Test the two solutions with red and blue litmus papers and record the changes you see.
    - c. Add two drops of phenolphthalein indicator to each test tube and record the changes you see.
    - d. Determine the pH of each solution using a pH meter. *Wash the electrode with dilute acetic acid and then distilled water to clean it between every reading and after you're done.*
  - 2. The Reaction of Metal Oxides and Water
    - a. In three test tubes, place 10 mL of water, 2 drops of phenolphthalein, and a pinch of calcium hydroxide, magnesium hydroxide, or calcium oxide. Record the color changes.
    - b. In this last section, you will explore the reaction that occurs when you heat limestone ("slaking lime") to make a compound known as "quicklime", which is used in the manufacture of concrete:

Take a small piece of iron wire and wrap it around a small chip of calcium carbonate (marble chip). Heat the chip until it is white hot with a Bunsen burner, for about 2 minutes. Let the chip cool and drop it into a beaker with 15 mL of water and a few drops of phenolphthalein. Compare this result to an unheated chip.

a	. •
Nec	f10n°
SUC	uon.

## **Data and Calculations for Experiment 9**

## A. Electrolytes and Instructor Demo

Place an "X" on the label that properly describes each compound below:

	Non- Electrolyte	Strong Electrolyte	Weak Electrolyte
1. Tap water			
2. Distilled water			
3. Sugar solution			
4. NaCl solution			
5a. Pure (glacial) acetic acid			
5b. Diluted acetic acid			
5c. Twice diluted acetic acid			
6a. 1 M acetic acid			
6b. 1 M HCl			
6c. 1 M NH4OH			
6d. 1 M NaOH			
7a. NaNO3			
7b. NaBr			
7c. Ni(NO <sub>3</sub> ) <sub>2</sub>			
7d. CuSO <sub>4</sub>			
7e. NH <sub>4</sub> Cl			

- 1. What reaction occurs when barium hydroxide and sulfuric acid are mixed?
- 2. Explain why the light becomes dimmer as two strong electrolytes are mixed with each other.
- 3. Why does the light come back on after more of the electrolyte is added?
- 4. What happens to the glacial acetic acid as it is diluted? How does this explain the changes in light intensity?

#### B. Properties of Acids

- 1. Reactions of Acids with Metals
  - a) Which acids reacted with the magnesium to produce H<sub>2</sub> gas?
  - b) Represent the reaction between the metal and ONE acid that occurred with an equation.
- 2. Measurement of pH and Acidity
  - a) Acids turned the red litmus paper \_\_\_\_\_\_.
  - b) Acids turned the blue litmus paper \_\_\_\_\_.
  - c) What is the color of phenolphthalein in acidic solution?
  - d) What is the pH of the 0.1 M solution?
    What is the pH of the 0.01 M solution
    What is the pH of the 0.001 M solution?
  - e) Which solution has the greatest concentration of  $H^+$ ?
  - f) Calculate the  $H^+$  concentration of a pH = 4.6 solution. Write the answer in scientific notation.
- 3. Reactions of Acids with Carbonates and Bicarbonates
  - a) What is the name and formula of the gas formed in this reaction?
  - b) What happened to the burning stick when it was placed in the beaker?

### Name: \_\_\_\_\_\_

#### Section:

c) Write out the products of the reactions in a balanced equation:

NaHCO<sub>3</sub> + HCl  $\rightarrow$ CaCO<sub>3</sub> + HCl  $\rightarrow$ 

- 4. Neutralizing Acids with Base: Using Indicators
  - a) Write a balanced equation for the reaction of HCl and NaOH.
  - b) What happened when the acid was all neutralized?
- 5. Reaction of a Non-Metal Oxide and Water
  - a) Write a balanced equation for the reaction of sulfur and oxygen.
  - b) What happens when the product of the above reaction reacts with water? Write a balanced equation that represents this reaction.
  - c) Write a balanced equation for the reaction of carbon dioxide and water.
  - d) How do you know that the product in the reaction above is acidic?

#### Name: \_\_\_\_\_

#### C. Properties of Bases

- 1. Properties of ammonium and sodium hydroxides
  - a) What did the sodium hydroxide feel like?
  - b) What did the ammonium hydroxide feel like?
  - c) Bases turned the red litmus paper \_\_\_\_\_.
  - d) Bases turned the blue litmus paper \_\_\_\_\_.
  - e) What is the pH of the ammonium hydroxide solution?
  - f) What is the pH of the sodium hydroxide solution?
  - g) Calculate the concentration of H<sup>+</sup> in the more basic solution
- 2. The Reaction of Metal Oxides and Water
  - a) What is the color of phenolphthalein with CaO?
    What is the color of phenolphthalein with MgO?
    What is the color of phenolphthalein with Ca(OH)<sub>2</sub>?
  - b) Write the balanced equations for the following reactions:

 $CaO \quad + \quad H_2O \quad \rightarrow \quad$ 

- $MgO \quad + \quad H_2O \quad \rightarrow \quad$
- c) Marble is calcium carbonate (CaCO<sub>3</sub>). Write a balanced equation for the reaction that occurs when you heat the marble chip.
- d) Write a balanced equation for the reaction that occurs when you put the heated marble chip in water.

# Experiment 10 – Lewis Dot Structures and Molecular Geometry

#### **Discussion**

The Valence Shell Electron Pair Repulsion (VSEPR) Theory states that bonds and lone pairs are regions of high electron density in an atom that repel each other until they get as far apart as possible. This effect determines the atom's geometry and bond angles. Two regions will be 180° apart, three regions will be 120° apart, and four regions will be 109.5° apart.

#### Geometry Determination

- A. Determine the Lewis dot structure of the molecule or ion.
- B. For each central atom in the structure, determine the areas of electron density that lie directly on that atom. An area of electron density may be:
  - a lone pair that lies on the central atom. (Lone pairs on other atoms don't count.)
  - a single bond.
  - a double bond.
  - a triple bond.
- C. Assign geometry according to the table on the next page.

#### **Procedure**

Do not build models for ionic compounds. For each of the polyatomic ions or molecules:

- II. Draw the Lewis dot structure, including all resonance where appropriate.
- III. Use the model kit to build the structure.
  - Use white or yellow balls for hydrogens.
  - Use black or blue or red balls for other atoms.
  - Use short sticks for nonbonded electron pairs (lone pairs).
  - Use long sticks for single bonds.
  - Use springs for double and triple bonds. Two springs form a double bond. Three springs form a triple bond.
- IV. Sketch the shape of the structure in three dimensions. This is called the VSEPR structure.
- V. Draw dipole moments on the VSEPR structure to show all polar bonds.
- VI. Give the name of the molecular geometry.
- VII. State whether the molecule is polar, nonpolar, or ionic.
- VIII. Determine the approximate bond angle on the central atom (if applicable).

Name: \_\_\_\_\_

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# of areas	# of bonds	# of lone pairs	Geometry and bond angles	Example
4	2	2	Angular or bent (109.5°)	H H
4	3	1	Pyramidal (109.5°)	H
4	4	0	Tetrahedral (109.5°)	
3	2	1	Bent (120°)	$\begin{bmatrix} \vdots & \vdots & \vdots \\ \vdots & \vdots & \vdots \\ 2 \text{ resonance forms} \end{bmatrix}$
3	3	0	Trigonal (120°)	$\begin{bmatrix} & & & & & & & & & & & & & & & & & & &$
2	2	0	Linear (180°)	$H - C \equiv N$
	1	any	Linear (Must be three or more atoms to form an angle.)	• <b>o</b> = <b>o</b>

Notice the convention for drawing bonds in 3-D space, where:

- the dash ( """"") represents a bond going behind the paper.

Bond angle on central atom(s)				
Polar? Nonpolar? Ionic?				
Molecular Geometry				
VSEPR structure (with dipole moments)				
Lewis dot structure (including ALL resonance)				
Number of valence electrons				
Formula	$\mathrm{I}_2$	NO <sup>-1</sup>	CO	BeH <sub>2</sub>

Section:

Bond angle on central atom(s)				
Polar? Nonpolar? Ionic?				
Molecular Geometry				
VSEPR structure (with dipole moments)				
Lewis dot structure (including ALL resonance)				
Number of valence electrons				
Formula	$H_2S$	PB <sub>r3</sub>	CIO4 <sup>-1</sup>	$CS_2$

Page 50

				I
Bond angle on central atom(s)				
Polar? Nonpolar? Ionic?				
Molecular Geometry				
VSEPR structure (with dipole moments)				
Lewis dot structure (including ALL resonance)				
Number of valence electrons				
Formula	CHC1 <sub>3</sub>	$PO_{3}$ -3	PO4 -3	$CH_2O$

Page 51

Name:			Section:	
Bond angle on central atom(s)				
Polar? Nonpolar? Ionic?				
Molecular Geometry				
VSEPR structure (with dipole moments)				
Lewis dot structure (including ALL resonance)				
Number of valence electrons				
Formula	SO <sub>3</sub>	SO <sub>3</sub> -2	SO4-2	SCN <sup>-1</sup>

Bond angle on central atom(s)				
Polar? Nonpolar? Ionic?				
Molecular Geometry				
VSEPR structure (with dipole moments)				
Lewis dot structure (including ALL resonance)				
Number of valence electrons				
Formula	NO <sup>2-</sup>	KCI	BrO <sub>3</sub> -	IO2 <sup>-</sup>

Chemistry M12 Laboratory Manual

Page 53

Bond angle on central atom(s)				
Polar? Nonpolar? Ionic?				
Molecular Geometry				
VSEPR structure (with dipole moments)				
Lewis dot structure (including ALL resonance)				
Number of valence electrons				
Formula	CH <sub>2</sub> Cl <sub>2</sub>	$CO_2$	$PH_3$	NaH

Chemistry M12 Laboratory Manual

# Experiment 11 – Boyle's Law

#### Discussion

Robert Boyle formalized his law around 1660. This is one of the earliest instances of the use of experiment and mathematics to describe physical systems as they are rather than using preconceived notions of what the system should be.

In this experiment, you will measure the pressure and volume of a sample of air trapped in a tube. Afterwards, you will examine the data and discover how close your data is to the ideal gas law.

Boyle's Law is simply stated as  $P \propto 1/V$ . That is, the pressure and volume are inversely proportional. Another way of writing this is PV = k. If the gas is "ideal", its pressure and volume will follow this equation at a constant temperature.

#### **Procedure**

- 1. Record the temperature (using a thermometer) and pressure (using a barometer) of the air in the room.
- 2. Obtain a gas pressure sensor, LabQuest mini, USB cable, chemistry department laptop computer, and a syringe.
- 3. Connect the gas pressure sensor to any channel on the LabQuest mini, then connect the USB cable from the LabQuest mini to the laptop computer
- 4. With the syringe disconnected from the pressure sensor, slide the plunger of the syringe so that the first black rubber gasket (the base of the rubber "cone") rests on the 10 mL line. Connect the syringe directly to the port on the pressure sensor with a gentle twisting motion do not over-tighten (see Figure 1). If the syringe or the pressure sensor has been damaged from overtightening (by a previous student), you may use Parafilm to form an air-tight seal between the syringe and the pressure sensor. For the remainder of the experiment, do not unscrew the syringe from the gas pressure sensor or remove the piston from the syringe. The air that is trapped inside the syringe will serve as the "gas" for your experiment.



Figure 1. Syringe connected to pressure sensor.

5. Open the Logger Pro software on the laptop computer. The computer monitor will display the pressure of the air that is trapped inside of the syringe, measured by the gas pressure sensor. This pressure is shown in the lower left corner of the Logger Pro window (see Figure 2). As you adjust the volume, you will record the pressure of the air inside the syringe in your data table in the column labeled "Pressure".



Figure 2. The Logger Pro window. In this example, the pressure is 99.00 kPa.

- 6. Gently depress the plunger until the first black gasket rests on the 5 mL line on the syringe. You will have to hold the plunger in. Do not push further than the 5 mL line – the maximum pressure the sensor can tolerate is 210 kPa.
- 7. Hold the plunger in this position for at least 15 seconds. If the pressure slowly drops during this time, then the seal between the syringe and the pressure sensor is not air-tight. Disconnect the syringe from the pressure sensor and go back to step 4! If the pressure is stable, then record the pressure in your data table.
- 8. Move the plunger to the 6 mL line on the syringe and hold it there. Record the pressure in the appropriate row of your data table. Repeat this procedure for each volume that is listed in your data table.
- 9. Open Microsoft Excel<sup>®</sup> on the laptop computer. Each of the "boxes" that you see are called "cells." In one row of cells, type the headings (in the same order) that you see at the top of your data table.
- 10. In the cells under the headings for "Syringe Volume (mL)" and "Pressure (kPa)," copy the numbers that you recorded in lab. Each cell should contain one number (without units).
- 11. Now, we are going to use Excel<sup>®</sup> to calculate the rest of our data for us. The volume printed on the side of the syringe does not account for the volume of air that is trapped in the tip of the syringe and inside of the gas pressure sensor itself. This volume has been measured to be 0.8 mL. To adjust for this volume, you will need to add 0.8 mL to each of the volumes listed in the data table. Click on the top empty cell under the "Actual Volume (mL)" heading. Type the equal sign (this tells Excel<sup>®</sup> to start a calculation), then click on the top

number in the "Plunger Position (mL)" column to use this number in your calculation. Type **+0.8** and hit enter. This cell should now display the number 5.8.

- 12. Repeat this process to calculate the top cell in each of the other columns. Be sure that you start each calculation by typing the equal sign and that use the actual volume (NOT the plunger position) for all of your calculations. "Click" on any number from the data table that will be a part of your calculation (do not enter any numbers by hand unless they are a part of a formula).
- 13. Once the first row of your data table is complete, click and drag to select the three cells that contain calculations. When these cells have been selected, a box will appear around them with a dark "dot" on the bottom-right corner (*Figure 3*). Double click on this dot and Excel<sup>®</sup> will automatically calculate all the rest of the rows in your data table.

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3	6.0	160.7						
4	7.0	140.1						
5	8.0	124.2						
6	9.0	111.5						
7	10.0	101.2						
8	11.0	92.6						
9	12.0	85.4						
10	13.0	79.2						
11	14.0	73.8						
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*Figure 3. A partially complete Excel*<sup>®</sup> *data table.* 

- 14. To calculate the average value of "k", find an empty cell away from your data table and type **=AVERAGE(** and then click and drag to select all of the numeric data in the  $P \times V = k$  (Pa·L) column. Press enter on the keyboard and then copy the average onto your data page.
- 15. Create two graphs using Excel<sup>®</sup>. On the first graph, you will need to graph Pressure vs. Actual Volume (this is Pressure on the y-axis, Volume on the x-axis). The first graph is not linear, so it should not have a trendline. On the second graph, you will need to graph Pressure vs. 1 / Volume (again Pressure on the y-axis, 1 / Volume on the x-axis). Add a linear trendline to the second graph and include the equation of the line. Be sure to save a copy of the Excel<sup>®</sup> data table and graphs to include with your report (or show them to your instructor before the end of lab).

### **Excel<sup>®</sup> Graphing Procedure**

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

Click and drag your mouse to highlight the boxes containing the numeric data for the x-axis. Then, *while holding CTRL on the keyboard*, click and drag your mouse to highlight the boxes containing the numeric data for the y-axis. From the "Insert" tab, choose a "Scatter" plot (*Figure 4*).

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Figure 4. Creating a Scatter Plot.

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

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

Section: \_\_\_\_\_

\_\_\_\_\_

Name: \_\_\_\_\_

## Data and Calculations for Experiment 11

Pressure of the air in the room:

Temperature of the air in the room:

Actual Volume	1 / Volume	Pressure * Vol.	Plunger	Pressure
(mL)	(mL <sup>-1</sup> )	$= k (Pa \cdot L)$	Position	(kPa)
			5	
	nes	e	6	
			7	
V		e	8	
	aulo	tod	9	
Cal	cula	leu	10	
in	Fvoo	R	11	
	LACC		12	
Sub	mit s		13	
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	j j j		18	
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8			20	

Average *k* = \_\_\_\_\_

### **Questions**

- 1) On your <u>linear graph</u>, do any points deviate from the straight line?
- 2) Write down the equation of the trendline (y = mx + b) from your linear graph. How does the slope (m) compare to the average P\*V=*k* value from the table of data?
- 3) Using the equation of your trendline, solve for the pressure at a volume of 2.0 mL. *Hint:* x = 1/V in your equation!
- 4) Why must the temperature be constant during this experiment? Use observations from your experiment and the graphs to support your answer!
- 5) If you repeated this experiment at a higher temperature, how would the P vs. V curve obtained differ from the curve on your 1<sup>st</sup> graph?
- 6) You have a 1.00 L sample of Argon gas at 700.0 mmHg. You decrease the pressure to 500.0 mmHg. What is the new volume?
- 7) Describe (quantitatively) what you would do to the volume of a container of gas if you wanted to double the pressure inside.

## Experiment 12 – Acid/Base Titrations

## Discussion

This experiment demonstrates an analytical technique known as titration, where a solution is delivered from a buret until it completely consumes another solution in a flask. Consider the following:

Acid-base titrations are an example of **volumetric analysis**, a technique in which one solution is used to analyze another. The solution used to carry out the analysis is called the **titrant** and is delivered from a device called a **buret**, which measures the volume accurately. The point in the titration at which enough titrant has been added to react exactly with the substance being determined is called the **equivalence point** (or **stoichiometric point**). This point is often marked by the change in color of a chemical called an **indicator**. The titration set-up is illustrated in the schematic shown.



First, the concentration of a base solution (standard) will be determined; this data will be used to determine the concentration of unknown acid solutions. To standardize the NaOH solution, it will be reacted with potassium hydrogen phthalate, KHC<sub>8</sub>H<sub>4</sub>O<sub>4</sub> (abbreviated KHP). The molar mass of KHP is 204.2 g/mol. The reaction of KHP with NaOH is known to be:

$$KHC_8H_4O_4(aq) + NaOH(aq) \rightarrow KNaC_8H_4O_4(aq) + H_2O(l)$$

Starting with a known mass of KHP and recording the volume of NaOH required to reach the endpoint, the molarity of the base can be determined. The indicator to be used, phenolphthalein, is colorless in acidic solution and rosy pink when slightly basic. Even though the endpoint is slightly overshot in order to make the color change, the goal is to use as little excess base as possible. Therefore, the titrated solution should be very pale pink, not bright rosy red, at the endpoint.

Once the concentration of the NaOH solution is known, one could determine the concentration of an acid solution by doing an additional titration.

### **Procedure**

- 1. Measure out 1.000 to 1.200 g of KHP and add to a 125 mL Erlenmeyer flask.
- 2. Add approximately 30 mL of D.I. water to the flask. If some KHP is sticking to the walls of the flask, rinse it down with D.I. water from a wash bottle.
- 3. Take a clean, dry 100 mL beaker to the hood and obtain approximately 60 mL of NaOH base. Be sure to keep the beaker covered with a watch glass when not in use.
- 4. Obtain a clean 50 mL buret. Carefully fill the buret with base, making sure that no air bubbles are present. Run some of the base solution through the buret tip to remove the air pocket in the tip.
- 5. Record the initial buret reading (x.xx mL) in the data section. When you read a buret, the line of sight must be level with the BOTTOM of the meniscus to avoid error. The top of the buret reads 0.00 mL; the bottom reads 50.00 mL. Notice the numbers increase going down. Take note of this when reading the numbers. Your instructor will demonstrate.
- 6. Add 2 to 3 drops of phenolphthalein indicator solution to the 125 mL Erlenmeyer flask containing KHP and water. Swirl your acid solution until the KHP is completely dissolved before moving on to the next step of the procedure.
- 7. Place the flask under the tip of the buret. A piece of white paper under the flask makes it easier to see the pale pink color at the endpoint. Open the valve and allow base to drip from the buret into the flask. Swirl continually to mix the solutions. As you get closer to the endpoint, the solution will begin to show pink color that goes away when you mix. Slow the rate of base addition to one drop at a time, mixing the solutions well after every drop. If you splash the solution up onto the sidewalls of the flask, spray a stream of water from your wash bottle over the inside of the flask. The extra water that mixes into your acid sample will not affect your results. Once the addition of ONE drop of base changes the solution from colorless to pale pink, close the buret valve and make certain that the pale pink color lasts for at least 30 seconds while swirling. If so, record the final buret reading. If not, carefully add one more drop of base from the buret valve until the pale pink color persists for 30 seconds of swirling or longer. If at the end of your trial the color is bright rosy red, you have overshot the endpoint. Make a note in your data if you overshoot.
- 8. Discard the titrated solution into the sink, rinse the flask with D.I. water, and then titrate another new sample of KHP following the same procedure above. Do at least TWO successful titrations that achieve a pale pink color of the indicator.
- 9. Calculate the average molarity of the base from your two successful trials and check with your instructor for verification.

## **Data and Calculations for Experiment 12**

	Sample 1	Sample 2
Mass of flask and KHP		
Mass of empty flask		
Mass of KHP		
Initial buret reading		
Final buret reading		
Volume of base used		

\_\_\_\_\_

1. Moles of acid (KHP, Molar mass = 204.2)

Sample 1:

Sample 2:

2. Moles of base used to neutralize acid

Sample 1:

Sample 2:

3. Molarity of base (NaOH)

Sample 1:

Sample 2:

4. Average Molarity of Base:

#### **Questions**

- 1. A titration required 13.42 mL of 0.1638 M NaOH solution. How many moles of NaOH were in this volume?
- 2. A student weighed a sample of KHP and found it weighed 1.396 g. Titration of this KHP required 21.36 mL of base (NaOH). Calculate the molarity of the base.
- 3. Write and balance the equation for the neutralization of a sulfuric acid solution of unknown concentration by sodium hydroxide. Calculate the molarity of an unknown sulfuric acid solution if a 25.0 mL sample of the acid solution consumes 27.2 mL of 0.138 M NaOH solution in a titration.

4. What might happen to your calculated NaOH molarity if you used tap water instead if D.I. water to dissolve the KHP crystals or to rinse down the walls of the flask during the titration? *Hint: Tap water contains some calcium carbonate*.

## Experiment 13 – Weighing by Difference

The purpose of this experiment is to learn how to correctly and accurately use the analytical mass balance.

### Procedure

NOTE: Always use the same balance during an experiment and leave it clean.

- A. Check to see if the balance is level by looking at the bubble in the level gauge. If the bubble is not centered, adjust the legs of the balance until the bubble is centered. Use the same balance throughout the experiment.
- B. Place a weighing boat on the balance pan. Zero the balance by pushing down on the tare button or zero button and waiting a few seconds before placing anything on it. Place approximately 3 grams of salt on the boat. Record the exact mass of the salt. Remove the weighing boat and salt from the balance and save them for step D.
- C. Zero the balance. Place a clean dry evaporating dish on the balance pan. Record its mass.
- D. Pour the salt from the weighing boat into the evaporating dish.
- E. Record the mass of the evaporating dish with the salt sample.
- F. Return the salt to its original container. Wipe clean and return the evaporating dish.

## Data and Calculations for Experiment 13

1. Mass of salt sample (from step B)	
2. Mass of evaporating dish	
3. Mass of evaporating dish and salt	
4. Mass of salt in evaporating dish (calculate)	
5. Difference between 1 and 4	

Show how you determined 4 and 5.

# Experiment 14 – Atomic Spectra

The purpose of this experiment is to show that different elements give off unique colors of light when atoms of the elements are excited by heating. By identifying the unique colors the element can be identified.

## <u>Part I</u>

There will be three gas discharge tubes set up in the lab. Observe the color of light given off by each discharge tube. Record the colors in the data table below. After recording the color of the light, observe the light through the diffraction grating (look off to one side). Draw a picture of the spectral lines. Identify the color of each line.

1.	Element		Color of light	
	Spectral diagram:			
		Violet		Red
2.	Element		Color of light	
	Spectral diagram:			
		Violet		Red
3.	Element		Color of light	
	Spectral diagram:			
		Violet		Red

## <u>Part II-A:</u>

There will be seven containers with wooden splints soaking in salt solutions. Each solution will be labeled with its chemical name.

### Procedure

Light the bunsen burner and adjust its flame until you see a blue inner cone. Use crucible tongs to remove a splint from the soaking solution and place it in the flame. Observe the color of the flame and record the color in the table below. If two solutions give colors which seem similar, repeat the experiment until you can notice the differences in color well enough that you can describe the differences. Always use the same burner with a given chemical. If you mix up burners, you can contaminate the colors.

Chemical	Color of Flame
lithium chloride	
calcium chloride	
potassium chloride	
copper(II) nitrate	
strontium chloride	
sodium chloride	
barium chloride	

## <u>Part II-B.</u>

There will be seven containers having unknown chemicals. Repeat the procedure that you used for known chemicals and identify the unknown chemicals. Record results below.

Unknown #	Color of Flame	Chemical
1		
2		
3		
4		
5		
6		
7		

## Experiment 19 – Specific Heat Capacity

The heat absorbed by any sample of matter when its temperature rises by 1  $^{\circ}$ C is called its heat capacity. Clearly, the heat capacity of a sample depends on its mass; the greater the mass of a sample, the greater the amount of heat it must absorb to increase its temperature by one temperature degree. The heat capacity of one gram of a substance is called its specific heat capacity, which is measured in J/g  $^{\circ}$ C or J/g K.

The molar heat capacity is the quantity of heat that must be absorbed by one mole of a substance to raise its temperature by 1 °C or 1 K. Its units are J/mol °C or J/mol K. The SI unit of energy is the Joule (J), where 4.184 Joules of heat are equal to 1 calorie. A calorie is defined as the energy required to raise 1 gram of water by 1 °C.

The relationship between the temperature change and the heat associated with the change is given by the equation:  $Q = ms\Delta T$ , where Q = heat, m = mass, s = specific heat, and  $\Delta T$  represents the change in temperature ( $T_{final} - T_{initial}$ ). In any experiment involving calorimetry, the amount of heat lost by the solid is equal to the amount of heat gained by the water. However, since heat loss is given a negative sign, we state that:

$$Q(gain) = -Q(loss)$$

This can also be represented by: Q(gain) + Q(loss) = 0.

## **Procedure**

- 1. Weigh and record the mass of a DRY metal sample. Please note that the mass of the attached string WILL NOT affect your results.
- 2. Place the loop of the string over an appropriate clamp to suspend the metal into a boiling D.I. water bath and allow the metal enough time to equilibrate to the temperature of the boiling water (about 5 minutes). Make certain that the metal is completely immersed at ALL times and does not touch the bottom of the beaker. After a constant temperature is observed, measure the temperature of the boiling water with a thermometer obtained from the lab cart, noting how to correctly read its scale. This is the initial temperature of the metal sample and should be recorded to within 0.01 °C. Afterwards, remove the thermometer from the water bath, and allow it to cool gradually.
- 3. Weigh an empty calorimeter (two styrofoam coffee-cups nested together with a plastic cover as the top). Use this same mass of the empty calorimeter for ALL TRIALS unless you use a different calorimeter.
- Add between 50 100 mL of water to the calorimeter (less for a small sample, more for a large sample). Weigh and record the mass of the calorimeter and water using the SAME analytical balance.

Name: \_

- 5. Place the styrofoam calorimeter into a glass beaker for added stability. Insert the thermometer through a hole in the calorimeter lid. Use a thermometer clamp to hold it in place while water equilibrates to a constant temperature (should remain constant for approximately 2 minutes). Occasionally swirl the water to obtain its average temperature. Record this equilibrium temperature to within 0.01 °C. Be careful not to place the calorimeter near the Bunsen burner.
- 6. Quickly and carefully place the hot metal into the water in the calorimeter. Carefully return the lid and thermometer to the calorimeter. Gently swirl the calorimeter while observing the temperature. Remove the thermometer from the clamp for this part and hold by hand so that the cup can be constantly swirled. Note the temperature immediately and continuously observe until a constant, HIGHEST stable temperature is reached. Record this final, highest temperature to within 0.01 °C.
- Repeat the experiment. Use the same calorimeter and piece of metal. Pour out the original water from the calorimeter and add a fresh 50 100 mL portion of water. Weigh and record this new mass. <u>Note</u>: If your temperature change was less than 1.2 °C, use less water in your second experiment.
- 8. When completely finished, dry the calorimeter, thermometer, and metal sample. Return them to the lab cart. Be sure to place the metal in its correct container.

Name:		Sec	ction:		
Data and Calculations					
	Trial 1		Trial 2		Trial 3
Mass dry calorimeter		=		=	
Mass calorimeter + volume H <sub>2</sub> O					
Initial temperature of water in calorimeter					
Mass of metal		=		=	
Initial temperature of hot metal (before adding it to calorimeter)					
Final temperature of water + metal in calorimeter					
$\Delta t_{water}$					
$\Delta t_{metal}$					

1. Calculate the specific heat of the metal from each trial and find the average value. If the two values do not agree to within 0.06 J/g °C, a third trial must be run. SHOW CALCULATIONS:

Trial 1	Trial 2	Trial 3

Average Specific Heat\_\_\_\_\_ J/g °C

2. Find the actual value for the specific heat of your metal in a reference book. Give this value in J/g °C or J/g K. Calculate the % error of your average value.

#### Post-lab Questions

- 1. Do objects that have the same temperature have the same amount of heat? Briefly explain.
- 2. What is the difference between something which is hot and something which has a lot of heat?
- 3. How much heat would it take to raise the temperature of 645 g of water by 25°C? SHOW CALCULATIONS.
- 4. When a 15.411 gram sample of metal gains 128.0 J of heat, its temperature changes from 18.55 °C to 83.00 °C. What is the specific heat of the metal? SHOW CALCULATIONS.

- 5. A metal sample weighing 71.9 g and at a temperature of 100.0 °C was placed in 41.0 g of water in a calorimeter at 24.5 °C. At equilibrium, the temperature of the water and metal was found to be 35.0 °C.
  - A. What was  $\Delta t_{water}$ ?
  - B. What was  $\Delta t_{\text{metal}}$ ?
  - C. How much heat flowed into the water?
  - D. Calculate the specific heat of the metal.

## Experiment 20 – Precipitation of Strontium Sulfate

In this experiment, you will study a precipitation reaction between sodium sulfate and strontium chloride. You will collect, dry, and weigh the precipitate and compare this experimental yield to the theoretical yield.

## **Procedure**

Weigh a clean, dry, 100-mL beaker. Add about 0.25 g (0.350 g max!) of solid sodium sulfate to the beaker and weigh it again. Dissolve the sodium sulfate in about 20 mL of D.I. water. Add 5.0 mL of 0.50 M strontium chloride solution and heat for fifteen minutes. Try to keep the mixture from boiling.

After the heating period has passed for the mixture, set it aside so as to return to room temperature, and then cool it further by putting the beaker in a cold water bath. Your precipitate should settle to the bottom, leaving a relatively clear solution above it. Obtain a piece of filter paper and weigh it on the analytical balance. Set up a vacuum filtration apparatus with a Büchner funnel and your weighed filter paper (your instructor will show you how). Using a stirring rod to guide the stream of liquid, pour the contents of the beaker into the Büchner funnel. Use your wash bottle (filled with D.I. water) to rinse any solid out of the beaker and into the filter. Make sure no precipitate remains in the beaker or on the stirring rod. Fill the beaker with 15 mL of D.I. water, swirl it around, and then pour it into the filter. Repeat the washing process, and then draw air through the funnel for a few minutes to help dry the crystals.

Turn off the vacuum, carefully remove the filter paper containing your precipitate with a spatula, and place it over a watch glass. Fill a 100-mL beaker half-way with water, place the watch glass with filter paper over the beaker, and heat to boil for twenty minutes to dry the precipitate (alternatively, you can place the watch glass with filter paper in a drying oven at 130 °C for twenty minutes). Allow to cool, then determine the mass of your precipitate. Heat for another five minutes, cool, and reweigh. The two weights should agree within  $\pm 0.05$  g or a third heating should be done.

#### **Data and Calculations for Experiment 20**

- 1. Weight of empty beaker
- 2. Weight of beaker and sodium sulfate
- 3. Weight of sodium sulfate

Show Calculation

Name: \_\_\_\_\_

Section:

4. Moles of sodium sulfate:

Show Calculation

5. Moles of strontium chloride moles  $\operatorname{SrCl}_2 = 5.0 \operatorname{mL} \operatorname{SrCl}_2 \left( \frac{10^{-3} \operatorname{L} \operatorname{SrCl}_2 \operatorname{solution}}{1 \operatorname{mL} \operatorname{SrCl}_2 \operatorname{solution}} \right) \left( \frac{0.50 \operatorname{mol} \operatorname{SrCl}_2}{1 \operatorname{L} \operatorname{SrCl}_2 \operatorname{solution}} \right) =$ 

Solve the Equation Shown

- 6. Write a balanced MOLECULAR equation for the reaction:
- 7. Write a balanced NET-IONIC equation for the reaction:
- 8. Weight of empty filter paper
- 9. Weight of filter paper and dried precipitate (first time)
  Weight of filter paper and dried precipitate (second time)
  Weight of filter paper and dried precipitate (third time)
  10. Weight of precipitate:

Show Calculation

11. Determine the limiting reactant and excess reactant for your reaction. Also, calculate the theoretical yield (in grams) of strontium sulfate.

Limiting Reactant: \_\_\_\_\_ Excess Reactant: \_\_\_\_\_

Show Calculation (theoretical product yield) Chemistry M12 Laboratory Manual 12. Determine the percentage yield of your reaction.

Show Calculation

13. Calculate the theoretical yield (in grams) of strontium sulfate if you had used half as much SrCl<sub>2</sub>(aq)?

Show Calculation

14. Calculate the theoretical yield (in grams) of strontium sulfate if you had used twice as much SrCl<sub>2</sub>(aq)?

Show Calculation

15. Briefly describe how you could have improved your percentage yield in this experiment.