

**Applied Chemistry** 

Chemistry 101

**Laboratory Manual** 

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## Syllabus

| Activity   | Report<br>Points | Unknown Points |
|--|------------------|----------------|
| Tour of the laboratory, Laboratory Procedures                                  |                  |                |
| Proper Use of Laboratory Notebook  |                  |                |
| Safety Video   |                  |                |
| Experiment #1: The Balance   | 10               |                |
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| Check In   |                  |                |
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| Experiment #4: Table Salt from Baking Soda                                     | 10               |                |
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| First Laboratory Exam  |                  |                |
| Experiments 1, 2, 3, 4, 5, and the Periodic Table Video                        |                  |                |
| You may consult your Laboratory Notebook                                       |                  |                |
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| Second Laboratory Exam   |                  |                |
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| You may consult your Laboratory Notebook                                       |                  |                |
| Experiment #13: Separation of Cations by Paper Chromatography                  | 10               | 15             |
| Experiment #14: Atomic Emission  |                  |                |
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| Experiment #15: The preparation and properties of NaHCO <sub>3</sub>           |                  |                |
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| Check out  |                  |                |
| Laboratory Final   |                  |                |
| Experiments 9, 10, 15, 41, 42, Temp and Crystal                                |                  |                |
| You may consult your Laboratory Notebook                                       |                  |                |
|  |                  |                |



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## **EXPERIMENT 1: The Balance**

## **PURPOSE**:

- 1. To learn to use the different types of balances which are available in the laboratory.
- 2. To learn the capabilities of the different types of balances which are available in the laboratory.
- 3. To relate the concept of significant numbers to the accuracy of mass and volume measurements.

## PRINCIPLES:

One of the most important operations in a chemistry laboratory is the massing of objects. Since chemistry is an exact science, the massing of substances which enter or result from a chemical change must be done with the best possible accuracy.

Balances differ in capacity and accuracy and the type of balance used in a particular experiment depends on the accuracy desired for that experiment.

For rough massings, where an accuracy of 0.1g is required, the platform decigram balance may be used. The centigram balance is conveniently used when an accuracy of 0.01g is required.

Semi-quantitative and some quantitative massing is commonly performed on the milligram balance, which reads to the nearest 0.001g The most accurate balances commonly used in the modern laboratories for accurate quantitative work are the analytical balances. While they are simple to use, they are also the most delicate and expensive.

The reliability of any balance depends upon how it is treated by the user, but special care is required in treating the analytical balance. For long balance life, certain general rules must be observed:

- 1. Keep the balance clean. Clean up any spills on, in, or near the balance, immediately.
- 2. Tare (zero) the balance prior to taking any measurements. Wait for the balance to indicate that it has been tarred prior to placing material on the balance.
- 3. Never place any chemical directly on the balance pan: always use a piece of weighing paper. Liquids must be weighed in a closed container.
- 4. When an analytical balance is used, the objects being massed should be handled with forceps or crucible tongs.
- 5. Objects being massed must always be at room temperature to avoid air currents forming which can affect the accuracy of the mass measurements. When using the analytical balance, always make sure that the windows of the balance are closed.

## PROCEDURE:

## 1. Mass of standard mass on an analytical balance:

Obtain a metal strip of known mass from your instructor and record its identification number. Determine its mass on the analytical balance and record the result. The mass you obtain should agree with the posted mass within 0.0005g. Calculate the Percent Error in your measurement by using the following formula:

Note: Typically the percent error has no sign (+/-); it is typically given as an absolute value.

## 2. The mass of a Penny:

Using the difference scales available; determine the mass of the same penny. Record these masses and indicate the accuracy in each measurement. State the number of significant figures in each measurement.

## 3. The density of a metallic cylinder



Obtain a metallic cylinder and record the material it composed of (brass, copper steel, or aluminum.) Using your ruler, measure the diameter (d) and the height (h) of the metallic cylinder to the nearest 0.1 mm. Calculate the volume of the metallic cylinder using the formula,  $V = \pi r^2 h$ . (Recall that 2r = d.)

Determine the mass of the cylinder using the centigram balance.

Calculate the density of the metallic cylinder. (Recall that d=m/V.)

Calculate the Percent Error in your density determination knowing that the theoretical values of the cylinder densities are:

| Brass:    | 8.50 g/cm <sup>3</sup> |
|-----------|------------------------|
| Copper:   | 8.96 g/cm <sup>3</sup> |
| Steel:    | 7.86 g/cm³             |
| Aluminum: | 2.70 a/cm <sup>3</sup> |

# Experiment 1: THE BALANCE REPORT FORM

| ME:                   |               | Date:                               | Partner(s                   | s):                              | · · · · · · · · · · · · · · · · · · · |
|-----------------------|---------------|-------------------------------------|-----------------------------|----------------------------------|---------------------------------------|
| Mass of Standa        | ard Mass or   | n Analytical Ba                     | lance                       |                                  |                                       |
| I.D. Number           | Exper         | imental Mass (g)                    | Theoretical I               | Mass (g)                         | % Error                               |
|                       |               |                                     |                             |                                  |                                       |
| Mass of Penny         |               |                                     |                             |                                  |                                       |
| Balance               | Mass (g)      | Number of<br>Significant<br>Figures | Number of<br>Certain Digits | Number of<br>Uncertain<br>Digits | Uncertainty (+/-) g                   |
| Decigram              |               |                                     |                             |                                  |                                       |
| Centigram             |               |                                     |                             |                                  |                                       |
| Milligram             |               |                                     |                             |                                  |                                       |
| Density of a Mo       |               | <u>ct</u>                           | _                           |                                  |                                       |
| Diameter: _           | (CI           | m) Radius: _                        | (cm)                        | Height:                          | (cm                                   |
| Volume:               | (CI           | m <sup>3</sup> )                    |                             |                                  |                                       |
| Mass:                 | (g            | )                                   |                             |                                  |                                       |
| Experimenta           | al Density: _ | (g/cm <sup>3</sup> )                |                             |                                  |                                       |
| Theoretical I         | Density:      | (g/cm <sup>3</sup> )                |                             |                                  |                                       |
| % Error<br>(show your | work):        | %                                   |                             |                                  |                                       |

| _ |   |   |    |    |   |   |   |   |
|---|---|---|----|----|---|---|---|---|
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| w | ч | • |    | LI | v |   | J |   |

Good laboratory techniques should provide you with a percent error of less than five (5) percent. If you obtained an error greater than five (5) percent, explain below what was the source of this error below. (If your error was less than five (5) percent, write "N/A" in the space provided.)
 If an analytical balance is available, why would you ever use the centigram balance?

3. Which of the balances used provided the greatest number of significant digits for mass of the penny?

### **Known Masses for Part 1**

K1 - 1.1877 g K2 - 0.9824 g K3 - 2.0557 g K4 - 2.0675 g K5 - 2.3550 g K6 - 1.5289 g K7 - 1.5957 g K8 - 1.2437 g

K9 - 1.6022 g K10 - 1.4881 g K11 - 1.8690 g

K12 – 1.6382 g K13 – 1.9364 g

K14 – 1.5274 g K15 – 1.6186 g

K16 – 1.2153 g K17 – 1.6696 g

K18 – 2.0222 g K19 – 1.7287 g

K20 – 1.9237 g

## **EXPERIMENT 2: Density**

## **PURPOSE**:

- 1. To provide practice with various measuring devices such as: rulers, balances, volumetric pipettes, and burets.
- 2. To collect data from which problems dealing with physical properties may be solved.
- 3. To calculate the density of various substances by measurements of length, volume and mass of objects.

## **PRINCIPLES**:

Density is a physical property of a substance and is often used as an aid to its identification. Density (D) is defined as the ratio of the mass (m) of a substance to the volume (v) occupied by that mass;

The units of density are:  $g/cm^3$  or g/mL (1 cm<sup>3</sup> = 1 mL)

While the mass of a substance is invariable, the volume occupied by the substance varies with the pressure and temperature to which it is subjected. Density therefore will also vary with pressure and temperature. The density of gases is affected by temperature and pressure more than liquids, while sol ids are affected the least. The effect of pressure on the density of liquids and solids is negligible for most considerations. The pressure effect will not be examined in this experiment.

## PROCEDURE:

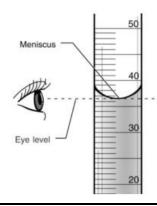
-----Day 1------

1. Density of glass.

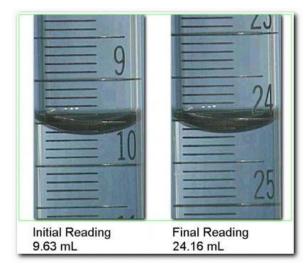
- a. Measure and record the mass of a glass rod on the centigram balance.
- b. Add approximately 20-25 mL of water to a 50 mL buret.
- c. Carefully read the liquid level at the bottom of the meniscus to the nearest 0.05 mL, making sure your line of sight is horizontal.

Note:

The correct way to read the volume of a liquid is to hold the graduated cylinder with the meniscus at eye level as shown in the drawing. Always read the level of the liquid at the bottom of the meniscus.



- d. Next, tilt the buret and carefully lower the glass rod to the bottom of the buret. Remove any air bubbles that may appear.
- e. Read and record the final water level in the buret.
- f. From this volume change, and the known mass, calculate the density of the glass rod and record it on your report form.



#### 2. Thickness of Aluminum Foil

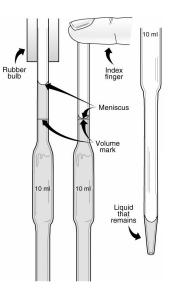
The volume of a rectangular solid is given by the product of its length, width and depth (thickness). If the object's density and mass are known, its thickness can be calculated if the length and width are measured.

- a. Obtain a rectangular or square sheet of aluminum foil (whichever is available in the laboratory).
- b. Measure the length and width of the foil to the nearest 0.05 cm, using a long ruler or a meter stick. Record your data on the report form.
- c. Fold the foil several times and weigh it on the analytical balance.
- d. Use your data and the density of aluminum (2.70 g/cm3) to calculate the thickness of your foil.
- e. Record the thickness of the foil in centimeters, meters and micrometers.

## -Day 2-----

## 3. Density of a liquid

The density of a liquid can be determined from its mass and an accurately measured volume using a volumetric pipette. A volumetric pipette is simply a glass tube with an enlarged barrel. The tip of the pipette is constricted and the upper part of the pipette tube has a calibration mark to which it is filled. When the pipette is filled and the liquid level (the bottom of the meniscus) is at the calibration mark, the pipette will deliver the indicated quantity of liquid.

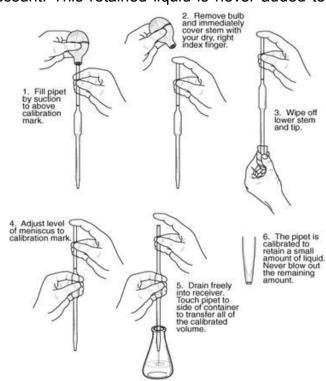


# NOTE: THE PIPET IS NEVER FILLED BY MOUTH SUCTION BUT ONLY BY USING A RUBBER SUCTION BULB

- a. Before use, the pipette must be clean. This can be checked by filling the pipette with water and allowing the liquid to drain. No water drop lets should be observed on the inner walls.
- b. Your volumetric pipette is calibrated TO DELIVER (TD) the indicated amount of liquid, e.g. 10.00 mL (to the nearest 0.02 mL), by gravity only. As the pipette drains, hold the tip of the pipette to the inner wall of the collecting vessel. When the flow of liquid from the pipette is complete, a small amount will remain in the tip. This type of pipette (TD) was calibrated taking this into account. This retained liquid is never added to

the amount of liquid delivered by gravity.

- c. The volume of a given amount of liquid will usually increase with an increase in temperature, e.g. the volume occupied by 1000 mL of water at 15°C will occupy 1002 mL at 25°C. For this reason pipettes are typically calibrated at a specific temperature (20°C).
- d. Obtain an unknown liquid sample from your instructor and record its number.
- e. Determine and record the



mass a clean dry empty vial and its cap on the analytical balance.

- f. Carefully pipette 10.00 mL of your unknown into the empty vial. Replace the cap on the vial.
- g. Determine and record the mass of the vial and its contents by using the analytical balance.
- h. Repeat the above procedure with a second 10.00 mL sample of your unknown.
- i. Calculate the density of the liquid for each of the two trials.
- j. Calculate the average of the two experimentally determined density values.

## Experiment 2: DENSITY REPORT FORM

NAME: \_\_\_\_\_ Date: \_\_\_\_\_ Partner(s):\_\_\_\_\_

| 1. | Density of Glass                   |          |          |          |
|----|------------------------------------|----------|----------|----------|
|    | Mass (g)                           |          |          |          |
|    | Initial Volume Reading (mL)        |          |          |          |
|    | Final Volume Reading (mL)          |          |          |          |
|    | Volume (mL)                        |          |          |          |
|    | Density (g/mL)                     |          |          |          |
| 2. | Thickness of Aluminum              |          |          |          |
|    | Length of Foil (cm)                |          |          |          |
|    | Width of Foil (cm)                 |          |          |          |
|    | Mass of Foil (g)                   |          |          |          |
|    | Thickness of Foil (cm)             |          |          |          |
|    | Thickness of Foil (m)              |          |          |          |
|    | Thickness of Foil (um)             |          |          |          |
| 3. | Density of a Liquid                |          |          |          |
|    | Unknown Number:                    |          |          |          |
|    |                                    |          | Sample 1 | Sample 2 |
|    | Mass of Vial and Cap (with liquid) | (g)      |          |          |
|    | Mass of Vial and Cap (without liqu | ıid) (g) |          |          |
|    | Mass of Liquid (g)                 |          |          |          |
|    | Volume of liquid (mL)              |          |          |          |
|    | Density of liquid (g/mL)           |          |          |          |
|    | Density of liquid (Average) (g/mL) |          |          | 1        |
|    |                                    |          |          |          |

| $\overline{}$ |   |   |    | ٠ |            |   |   |   |
|---------------|---|---|----|---|------------|---|---|---|
| Q             | 1 | Ω | ct | 1 | $^{\circ}$ | n | c | • |
|               | w | 7 | อเ |   | u          |   | o | _ |

| \ues | tions:  |
|------|---|
| 1.   | Calculate the percentage error of your experimentally determined density of glass. (Assume that the density of glass is equal to 2.5 g/mL.) |
|      | glade. (Aleeanne that the density of glade to equal to 2.0 g/m2.)   |
|      |   |
|      |   |
|      |   |
|      |   |
|      |   |
|      |   |
|      |   |
|      |   |
|      |   |
| 2.   | Why should you or shouldn't you blow all of the liquid out of the pipette in section 2?   |
|      | 2!  |
|      |   |
|      |   |
|      |   |
|      |   |
|      |   |
|      |   |

# **EXPERIMENT 3: Determination of the Empirical Formula of a**Compound

## **PURPOSE:**

1. To determine the empirical formula of a metallic oxide.

## **PRINCIPLES:**

The empirical formula gives the relative numbers of the different kinds of atoms which are present in a compound. We can determine the relative weight of the different elements in a compound if we can synthesize the compound from its elements or analyze the compound to obtain the constituent elements. From knowledge of the atomic weights of the different elements, we can determine the relative number of atoms (or moles of atoms) in a given mass of the compound. By combining these Ideas, we can see that in a compound which contains x grams of element X and y grams of element Y:

the number of moles of atom 
$$X = \frac{x \text{ grams}}{Atomic \text{ Mass of } X}$$

the number of moles of atom 
$$Y = \frac{y \text{ grams}}{Atomic \text{ Mass of } Y}$$

The relative number of moles of atoms of the elements would then be the ratio of X to Y. In writing the empirical formula we reduce the ratios of the relative numbers of atoms to the ratio of the smallest whole numbers, and use the smallest whole numbers in writing the empirical formula.

The relative masses of the different elements in a compound can be obtained by a variety of techniques.

In the present experiment, we will oxidize tin with nitric acid and then heat the product so that only tin oxide remains. The reaction can be abbreviated as:

$$Sn \xrightarrow{HNO_3} SnO_x$$

where x is the key number if the empirical formula is to be determined in this experiment.

If we know the initial mass of the tin used and the mass of the oxide produced, then the mass of the oxygen present is given by the increase in the mass of the sample. From this data we can then proceed to determine the empirical formula of the tin oxide which is produced.

#### **SAFETY PRECAUTIONS:**

The concentrated nitric acid used in this experiment should be kept and used in the hood. The reaction of tin with nitric acid gives off fumes containing nitrogen oxides which should not be inhaled. Protective safety glasses should be worn and the nitric acid should not be dropped or splattered on oneself.



## GOGGLES must be worn throughout this experiment.

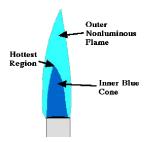


### PROCEDURE:

- 1. First you must clean and dry your crucible.
  - a. Wash the crucible and wipe it dry.
  - b. Place the crucible on a clay triangle and heat it gently
  - c. Heat for two or three minutes at maximum flame temperature. The tip of the sharply defined inner blue cone of the flame (hottest part) should almost touch the crucible bottom.



- d. Transfer the crucible, using the crucible tongs, to a clean, dry, heat-resistant surface to cool.
- 2. Allow the crucible to cool for a minimum of five minutes.
- 3. Mass and record the crucible's mass to the nearest 0.0001 g.
- 4. Place a piece of tin foil massing about 1 gram into your crucible.



- 5. Mass and record the mass of the crucible plus the foil to the nearest 0.0001 g.
- 6. Calculate the exact mass of the tin used.
- 7. Place the crucible into the hood and add concentrated nitric acid drop wise to the tin in the crucible. NOTE: The crucible shall not be heated during the addition of the HNO<sub>3</sub>.
  - a. Observe the reaction cautiously after the addition of each portion of concentrated nitric acid.
  - b. Do not add the nitric acid so fast that it foams or splatters out of the crucible.
- 8. After the tin has completely reacted with the nitric acid, the evolution of brown fumes (nitrogen dioxide) will stop.
  - a. When this stage has been reached, the crucible should be placed in the clay triangle on the ring stand in the hood.
  - b. Warm gently the crucible and its contents to evaporate any excess unreacted nitric acid.

c. No more brown fumes of nitrogen dioxide are given off when the evaporation is complete.



Warning: Do not heat the crucible strongly before all of the nitric acid has been evolved because the sample may splatter. Any splattering will result in an increased error in your final calculations.



- d. Heat for a minimum of five minutes at maximum flame temperature. The tip of the sharply defined inner blue cone of the flame (hottest part) should almost touch the crucible bottom.
- e. Transfer the crucible, using the crucible tongs, to a clean, dry, heat-resistant surface to cool.
- f. Allow the crucible to cool for a minimum of five minutes
- 9. Mass and record the crucible and its contents to the nearest 0.0001 g.
- 10. Heat the crucible and contents for a minimum of five minutes at maximum flame temperature.
- 11. Transfer the crucible, using the crucible tongs, to a clean, dry, heat-resistant surface to cool.
- 12. Allow the crucible to cool for a minimum of five minutes
- 13. Mass and record the crucible and its contents to the nearest 0.0001 g.
  - a. If the two masses do not agree within 0.0005 g. repeat the heating process again until constant mass is achieved.

#### CALCULATIONS:

- 1. Subtract the mass of the crucible plus tin from the mass of crucible plus tin oxide (constant mass) to find the mass of the oxygen in the tin oxide.
  - NOTE: Constant mass is always taken as the lowest mass.
- 2. Divide the mass of tin by its atomic weight to get the relative number of moles of tin atoms. Do the same for oxygen. Reduce the relative numbers of moles to a whole number ratio. Round off the answer to the simplest whole number.
- 3. Record the empirical formula of the tin oxide you have synthesized.

## **CLEANING UP:**

1. The crucibles may be cleaned out by scraping out the loose tin oxide and dissolving any that sticks to the crucible with a few drops of concentrated hydrochloric acid.

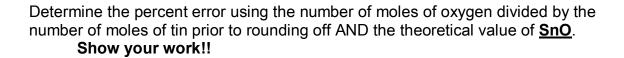


CAUTION!!! DO THIS IN THE HOOD



# Experiment 3: Determination of the Empirical Formula of a Compound REPORT FORM

| NAME:                         | Date:        | Partner(s):       |
|-------------------------------|--------------|-------------------|
|                               |              |                   |
| Mass of crucible:             |              | g                 |
| Mass of crucible + tin:       |              |                   |
| Mass of tin:                  |              |                   |
| Massif crucible + tin oxide:  |              | g (Constant Mass) |
| Mass of crucible + tin:       |              |                   |
| Mass of oxygen in oxide:      |              |                   |
| Relative number of moles of S | n atoms:     | moles             |
| Mass of 1 mole of Sn:         |              | g / mole          |
| Relative number of moles of O | atoms:       | moles             |
| Mass of 1 mole of O:          |              |                   |
|                               | Actual Value | Rounded Off Value |
| Number of moles of Sn _       |              |                   |
| Number of moles of Sn         |              |                   |
| Number of moles of O _        |              |                   |
| Number of moles of Sn =       |              |                   |
| Empirical Formula of Oxide Fo | ormed        |                   |



Determine the percent error using the number of moles of oxygen divided by the number of moles of tin prior to rounding off AND the theoretical value of <u>SnO</u><sub>2</sub>. **Show your work!!** 

## **EXPERIMENT 4: Table Salt from Baking Soda**

## **PURPOSE:**

- 1. To obtain sodium chloride from sodium hydrogen carbonate
- 2. To study the stoichiometry of this reaction

## **METHOD:**

Baking soda is the common name for sodium hydrogen carbonate [NaHCO<sub>3</sub>]. When sodium hydrogen carbonate is treated with hydrochloric acid [HCl<sub>(aq)</sub>] it produces a white solid residue (sodium chloride, commonly called table salt) and two gaseous products: water vapor and carbon dioxide.

## GOGGLES must be worn throughout this experiment.

### PROCEDURE:

- 1. Mass a clean, dry, 50 mL beaker on the centigram balance.
- 2. Remove the beaker from the balance and add approximately 1.0g-1.5g of NaHCO<sub>3</sub> into the beaker.



### DO NOT EXCEED THE AMOUNT OF NaHCO<sub>3</sub> INDICATED.

- 3. Determine the exact mass of the beaker and its content on the centigram balance. The exact mass of the NaHCO<sub>3</sub> in the beaker may be determined by difference of this and the empty beaker.
- In the fume hood, measure out 4-5 mL of concentrated hydrochloric acid (12 M) in your small graduated cylinder. Record this volume to the nearest 0.1 mL (You must measure out at least 4.0 mL).



## CAUTION!! CONCENTRATED HYDROCHLORIC ACID IS HIGHLY CORROSIVE AND GIVES OFF NOXIOUS FUMES!



5. Transfer the concentrated hydrochloric acid to a small beaker and cover it with a watch glass. Leave the beaker under the fume hood.

DO NOT RETURN ANY CONCENTRATED HYDROCHLORIC ACID TO THE ORIGINAL REAGENT BOTTLE.

DO NOT REMOVE THE CONTAINER WITH HYDROCHLORIC ACID FROM THE FUME HOOD.

If you measured out too much concentrated hydrochloric acid, dispose of it in an appropriately labeled waste container found in the fume hood.

6. While In the fume hood, add drop wise (use a Pasteur pipette) the concentrated hydrochloric acid to the sample and observe the effervescence. If the effervescence is too vigorous, slow down the rate at which the concentrated hydrochloric acid is added, to avoid splattering of the sample.

This step is completed when all of the concentrated hydrochloric acid has been added to the sample.

7. Heat the beaker under the fume hood until the sample appears dry.

If the residue starts to melt (glassy appearance), this indicates that the heating is too strong and the residue had probably been already heated to dryness and hence constant mass. Keep in mind that heating is done with the sole purpose to completely drive off the gaseous products, and not to melt the residue.

- 8. Continue heating at in the hood until constant mass is achieved. (Constant mass will occur when successive massings agree within 0.01 g)
- 9. Record the mass of the beaker and the residue (constant mass) and determine the mass of the residue.

## Experiment 4: Table Salt from Baking Soda REPORT FORM

| NAME:                 | Date:  | Partner(s): |        |       |
|-----------------------|--|-------------|--------|-------|
| <u>Data</u>           |  |             |        |       |
| Mass of beaker:       |  |             | g      |       |
|                       | ample (NaHCO <sub>3</sub> ):                     |             |        |       |
| Mass of sample (NaH   | CO <sub>3</sub> ):                               |             | g      |       |
| Mass of beaker and re | esidue first heating:                            |             | g      |       |
| Mass of beaker and re | esidue second heating:                           |             | g      |       |
| Mass of beaker and re | esidue third heating (if requ                    | uired):     | g      |       |
| Mass of residue:      |  |             | g      |       |
| Volume of concentrate | ed HCl added:                                    |             | mL     |       |
| <u>Calculations</u>   |  |             |        |       |
| Mass of 1 mole of Nal | HCO <sub>3</sub> :                               |             | g/mole |       |
| Number of moles of N  | aHCO₃ added:                                     |             | moles  |       |
| Concentration of HCI  | added:   |             | M      |       |
| Number of moles of H  | CI   |             | moles  |       |
|                       | emical equation for this reactants and products. |             |        | state |
|                       | HCI are required to react                        |             |        | have  |

| Which reactant is limiting?  |   |
|------------------------------|---|
| Which reactant is in excess? |   |
| Actual yield of NaCl:        | g |
| Theoretical yield of NaCl:   | g |
| Show calculations below:     |   |
|                              |   |
|                              |   |
|                              |   |
|                              |   |

Percent yield of NaCl \_\_\_\_\_\_\_ %

Show calculations below:

Chemistry 101

## **EXPERIMENT 5: Analysis of a Mixture of NaHCO<sub>3</sub> and NaCl**

## **PURPOSE:**

- 1. To determine the percent of NaHCO<sub>3</sub> in a mixture of NaHCO<sub>3</sub> and NaCl.
- 2. To separate two substances by a chemical reaction.

## **METHOD:**

When a mixture of NaHCO<sub>3</sub> and NaCl is treated with concentrated HCl, only NaHCO<sub>3</sub> will react, whereas the NaCl will remain unchanged:

$$NaHCO_{3(s)} + HCl_{(aq)} \rightarrow NaCl_{(s)} + CO_{2(g)} + H_2O_{(g)}$$
  
 $NaCl_{(s)} + HCl_{(aq)} \rightarrow No \ reaction$ 

The sample of unknown contains two compounds (NaHCO<sub>3</sub> and NaCl). The residue which results upon addition of aqueous HCl will contain only NaCl. Part of the residue is the original NaCl that was present and the other part is the NaCl formed as a result of the reaction between NaHCO<sub>3</sub> and HCl.

#### Since:

- The mole ratio between the NaCl formed and the NaHCO<sub>3</sub> reacted is 1:1 (e.g., 1 mole of NaHCO<sub>3</sub> produces 1 mole of NaCl)
- The molar mass of NaCl is less than the molar mass of NaHCO<sub>3</sub>
- The other products of the reaction (CO<sub>2</sub> and H<sub>2</sub>O) are gases and will be lost to the surroundings.

Then the residue will weigh less than the original sample and the sample will appear to have "lost mass".

Since the mass loss is proportional to the mass of NaHCO<sub>3</sub> originally present in the mixture, the mass of NaHCO<sub>3</sub> can be calculated. The result of this analysis is usually given as the percentage of NaHCO<sub>3</sub> present in the original sample of unknown composition.

## PROCEDURE:

- 1. Obtain a sample (about 1.0 grams) of the unknown (NaCl + NaHCO<sub>3</sub>) mixture.
- 2. Record the unknown number in your Laboratory Notebook.
- 3. Place a clean 50 mL beaker on a wire gauze and heat for three minutes at maximum flame temperature to remove all moisture from the beaker.



- 4. Allow the beaker to cool to room temperature.
- 5. Mass the cooled beaker on the analytical balance (to the nearest 0.0001 g).
- 6. Add your sample into the beaker.
- 7. Determine the exact mass of the beaker and its contents on the analytical balance (to the nearest 0.0001 g).
- 8. Determine the exact mass of the sample in the beaker by difference.
- 9. In the fume hood, measure out 4-5 mL of concentrated hydrochloric acid (12 M) in your small graduated cylinder.



## CAUTION!! CONCENTRATED HYDROCHLORIC ACID IS HIGHLY CORROSIVE AND GIVES OFF NOXIOUS FUMES!



10. Record the volume of the acid to the nearest 0.1 mL

NOTE: YOU must measure out at least 4.0 mL of the acid. This will ensure that the HCl is NOT the limiting reactant.

11. In the fume hood, Transfer the concentrated hydrochloric add to <u>another</u> small beaker and cover it with a watch glass.



DO NOT RETURN ANY OF THE CONCENTRATED HYDROCHLORIC ACID TO THE ORIGINAL REAGENT BOTTLE.





## DO NOT REMOVE THE CONTAINER WITH HYDROCHLORIC ACID FROM THE FUME HOOD



- 12. If you measured out too much concentrated hydrochloric acid, dispose of it into an appropriately labeled waste container found in the fume hood.
- 13. While in the fume hood, add by drops using a Pasteur pipette the concentrated hydrochloric acid to the sample and observe the effervescence.

NOTE:

If the effervescence is too vigorous, slow down the rate at which the concentrated hydrochloric acid is added. This is to avoid splattering of the sample. This step is completed when all of the concentrated hydrochloric acid has been added to the sample.

14. Heat the beaker in the fume hood until the sample appears dry.

NOTE:

Avoid strong heating, especially at the beginning since the excess hydrochloric acid may boil too vigorously. This will cause loss of the residue and poor experimental results. Evaporation should occur but not too strong as to cause loss of residue (splattering.) The easiest way to control the heating is by moving the Bunsen burner with your hand.

15. Continue to heat the beaker for an additional five minutes.

NOTE:

If the residue starts to melt (glassy appearance), the heating is too strong and the residue had probably been already heated to dryness. Heating is done with the purpose to drive off the gaseous products, and not to melt the residue.

Under no circumstances should you heat the residue with a strong flame (crucible bottom is red) for more than 5 minutes.

- 16. Allow the beaker to cool to room temperature
- 17. Determine the mass of the beaker and its contents on the analytical balance as soon as the beaker reached room temperature.

NOTE:

If the residue of NaCl is kept for a while in the open air, it will absorb moisture from the air, which will add to the mass of the residue and result in an incorrect final value.

- 18. Return the beaker and contents to the triangle and reheat the beaker strongly (beaker bottom will turn red) for 3 minutes.
- 19. Allow the beaker to cool to room temperature
- 20. Determine the mass of the beaker and its contents on the analytical balance as soon as the beaker reached room temperature.
- 21. If the mass of the beaker and its contents has changed by more than 1 mg (0.0001 g) from the pervious heating, reheat until a constant mass is obtained.



Blank Page

# Experiment 5: Analysis of a Mixture of NaHCO<sub>3</sub> and NaCl REPORT FORM

| NAME:                                | Date:                          | Partner(s): |    |
|--------------------------------------|--------------------------------|-------------|----|
| UNKNOWN NUMBER:                      |                                |             |    |
| Data:                                |                                |             |    |
| Mass of beaker and sample            | (NaHCO <sub>3</sub> + NaCl): _ |             | g  |
| Mass of beaker:                      |                                |             | g  |
| Volume of 12M HCl added: _           |                                |             | mL |
| Mass of beaker and residue           | 1 <sup>st</sup> heating:       |             | g  |
| Mass of beaker and residue           | 2 <sup>nd</sup> heating:       |             | g  |
| Mass of beaker and residue           | 3 <sup>rd</sup> heating:       |             | g  |
| Mass of beaker and residue           | 4 <sup>th</sup> heating:       |             | g  |
| Mass of beaker and residue           | BEST heating:                  |             | g  |
| Mass of sample (NaHCO <sub>3</sub> + | NaCI)                          |             | g  |
| Mass of residue (NaCl)               |                                |             | g  |
| Mass lost by sample                  |                                |             | 0  |
| wass iost by sample                  |                                |             | g  |

## **Calculations:**

Assume that 1.000g of NaHCO $_3$  is reacted with excess HCI and completely converted to NaCI. Calculate the mass of NaCI obtained.

## Show your calculations below:

| Mass of NaHCO <sub>3</sub> :                                     | 1.000 | <u>_</u> g |
|--|-------|------------|
| Mass of NaCl obtained:   |       | <u></u> g  |
| Mass lost by 1.000g of NaHCO <sub>3</sub> :(Calculated)          |       | <u>_</u> g |
| Experimental Data:   |       |            |
| Mass lost by sample:(Experimental, see first page)               |       | <u>_</u> g |
| Mass of NaHCO <sub>3</sub> in original sample:                   |       | <u></u> g  |
| Mass of sample:  |       | g          |
| Percent of NaHCO <sub>3</sub> in original sample(Show your work) |       | _ %        |

## **EXPERIMENT 6: Net Ionic Equations**

## **PURPOSE:**

- 1. To distinguish between an overall equation, a total ionic equation and a net ionic equation.
- 2. To write an overall equation, a total ionic equation and a net ionic equation for reactions where the reactants yield:
  - a. A precipitate
  - b. A gas
  - c. An un-ionized product
  - d. No reaction

### PRINCIPLES:

The most common method to represent a chemical reaction is a **balanced chemical equation**. This equation is commonly referred to as an **overall** or **molecular** equation. This equation describes the chemical reaction in general terms, but it may not describe the precise chemical changes that occur at the molecular or ionic levels. This is because the species that actually experiences the chemical change may be an ion, which is only part of the compound from which it is obtained.

In order to distinguish between substances that are present as ions in solution and those that exist as molecules or as ionic solids, an *ionic equation* is written.

A **total ionic equation** includes all chemical species actually present at the scene of a reaction except the solvent water, which is neither a reactant nor a product, but merely the medium in which the reaction occurs.

Not all species at the reaction scene undergo chemical change. Substances which are present but experience no chemical change are called **spectators**.

A **net ionic equation** is an equation in which spectators are removed from the total ionic equation. The net ionic equation shows only those reactants that are actually consumed and those products that are actually formed in the reaction.

In this experiment you will direct your attention to writing net ionic equations. In net ionic equations it is necessary to clearly show the state of a reactant or product by writing the state designation. The state designations are written after the formula of the species in the equation.

| (s) Solids                | i.e. H <sub>2</sub> O <sub>(s)</sub> |
|---------------------------|--------------------------------------|
| (I) Liquids               |                                      |
| (g) Gases                 |                                      |
| (aq) for aqueous solution | i.e. NaCl <sub>(aq)</sub>            |

In order to represent chemical compounds correctly in ionic equations, one must recognize whether a compound will be present in ionic or molecular form.

The following criteria may be used to determine the state of a species:

### 1. Strong electrolytes in solution are always present in the form of aqueous ions.

Strong electrolytes include:

## a. Strong acids:

The common strong acids are:

### b. Strong bases:

Strong bases are the hydroxides of the alkali (Group IA) and alkaline earth (Group IIA) metals which are sufficiently soluble.

The common strong bases are:

LiOH ...... Lithium hydroxide

NaOH ..... Sodium hydroxide

KOH ..... Potassium hydroxide

Ba(OH)<sub>2</sub> ..... Barium hydroxide

Sr(OH)<sub>2</sub> ..... Strontium hydroxide

Ca(OH)<sub>2</sub> ..... Calcium hydroxide

#### c. Soluble Salts:

Determinations of the solubility of a salt may be made by reference to the SOLUBILITIES OF IONIC COMPOUNDS table given on the next page.

|                  | SOLUBILITIES OF IONIC COMPOUNDS              |                 |                 |   |     |                   |                                |                                |                                |                 |                               |
|------------------|--|-----------------|-----------------|---|-----|-------------------|--------------------------------|--------------------------------|--------------------------------|-----------------|-------------------------------|
| lons             | C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> | CI <sup>-</sup> | Br <sup>-</sup> | ľ | OH. | NO <sub>3</sub> - | SO <sub>4</sub> <sup>2</sup> - | SO <sub>3</sub> <sup>2</sup> - | CO <sub>3</sub> <sup>2</sup> - | S <sup>2-</sup> | PO <sub>4</sub> <sup>3-</sup> |
| Na⁺              | s  | s               | S               | s | s   | S                 | S                              | S                              | s                              | S               | s                             |
| K⁺               | s  | S               | S               | S | s   | s                 | s                              | S                              | s                              | s               | S                             |
| Ag⁺              | Р  | ı               | ı               | ı | -   | s                 | Р                              | I                              | ı                              | I               | I                             |
| NH4 <sup>+</sup> | s  | S               | S               | s | -   | S                 | S                              | s                              | S                              | S               | s                             |
| Fe <sup>2+</sup> | s  | S               | S               | s | ı   | s                 | s                              | I                              | ı                              | S               | I                             |
| Ni <sup>2+</sup> |  | S               | S               | S | ı   | s                 | s                              | ı                              | ı                              | ı               | I                             |
| Mg <sup>2+</sup> | s  | S               | S               | S | ı   | s                 | s                              | S                              | ı                              | -               | I                             |
| Ca <sup>2+</sup> | s  | S               | S               | s | Р   | S                 | Р                              | Р                              | ı                              | ı               | I                             |
| Ba <sup>2+</sup> | -  | S               | S               | s | Р   | Р                 | ı                              | ı                              | ı                              | -               | I                             |
| Cu <sup>2+</sup> | Р  | S               | -               |   | ı   | S                 | S                              |                                |                                | ı               | I                             |
| Pb <sup>2+</sup> | s  | Р               | Р               | ı | ı   | S                 | ı                              |                                | ı                              | I               | I                             |
| Zn <sup>2+</sup> | s  | S               | s               | S | I   | S                 | S                              | I                              | I                              | I               | I                             |
| Al <sup>3+</sup> | Р  | S               | S               | - | I   | S                 | S                              |                                |                                | -               | I                             |
| Fe <sup>3+</sup> | -  | S               | S               | - | I   | S                 | S                              |                                |                                | -               | I                             |

A "S" indicates a soluble species

## 2. Weak electrolytes in solution are always present in molecular form.

Weak electrolytes or un-ionized substances include:

- a. Weak acids
- b. Weak Bases
- c. Water

A "I" indicates an insoluble species

A "-" (dash) indicates an unstable species A " " (blank) indicates lack of data

## 3. Insoluble substances are always present as the total compound: their chemical formulas are written accordingly.

These Substances include:

- a. Precipitates
- b. Gases

#### 4. Unstable substances are written in the form of their decomposition products.

There are three frequently encountered unstable products. When these are formed in chemical reactions they decompose immediately to form new products.

a. Sulfurous acid:

$$H_2SO_{3(aq)} \rightarrow SO_{2(g)} + H_2O_{(I)}$$

b. Carbonic acid:

$$H_2CO_{3(aq)} \rightarrow CO_{2(g)} + H_2O_{(l)}$$

c. Ammonium hydroxide:

$$NH_4OH_{(aq)} \rightarrow NH_{3(aq)} + H_2O_{(l)}$$

#### 5. The net ionic equation must be balanced both atomically and electrically.

Since a net ionic equation contains ions which are electrically charged, the total charge on either side of the equation is not necessarily zero. Just as the number of atoms must be the same on both sides of an equation, so also must the algebraic sum of the charges of all ions or molecules (or both) must be the same on both sides of an equation.

From these principles, we derive a three-step approach to writing net ionic equations:

#### 1. Write the overall equation showing the formulas of the compounds involved.

- a. Where products are not given, keep in mind that most of the react ions are double replacement react ions. This will help you in predicting the products formed.
- b. Determine the states of all of the reactants and products from the five criteria given above.
- c. Complete the state designations for all reactants and products.
- d. Balance this equation.

#### 2. Write the total ionic equation.

- a. Determine whether dissolved species are aqueous ions (strong electrolytes) or dissolved molecules (weak or non-electrolytes).
- b. Write the dissolved species in the form in which they are actually present.
- c. If you keep the coefficients from the overall equation, this equation will also be balanced.

- 3. Write the net ionic equation.
  - a. Eliminate spectator ions from the total ionic equation and rewrite the equation.
  - b. This equation must also be balanced.

## **EXAMPLES**:

- 1. Hydrobromic acid and a solution of ammonium carbonate are combined.
  - a. Overall equation:

$$2HBr_{(aq)} + (NH_4)_2CO_{3(aq)} \rightarrow 2NH_4Br_{(aq)} + H_2CO_{3(aq)}$$
  
Strong Acid Soluble Salt Soluble Salt Unstable Substance

b. Overall ionic equation:

$$2H^{+}_{(aq)} + 2Br^{-}_{(aq)} + 2NH_{4}^{+}_{(aq)} + CO_{3}^{2-}_{(aq)} \rightarrow 2NH_{4}^{+}_{(aq)} + Br^{-}_{(aq)} + H_{2}O_{(l)} + CO_{2(g)}$$

c. Net ionic equation:

$$2H^{+}_{(aq)} + CO_{3}^{2-}_{(aq)} \rightarrow H_{2}O_{(l)} + CO_{2(g)}$$

- 2. A solution of potassium acetate is added to nitric acid.
  - a. Overall equation:

$$KC_2H_3O_{2(aq)} + HNO_{3(aq)} \rightarrow KNO_{3(aq)} + HC_2H_3O_{2(aq)}$$
  
Soluble Salt Strong Acid Soluble Salt Weak Acid

b. Overall ionic equation:

$$K^{+}_{(aq)} + C_2 H_3 O_2^{-}_{(aq)} + H^{+}_{(aq)} + NO_{3(aq)} \rightarrow K^{+}_{(aq)} + NO_3^{-}_{(aq)} + HC_2 OH_3 O_{2(aq)}$$

c. Net ionic equation:

$$C_2H_3O_2^{-}{}_{(aq)} + H^{+}{}_{(aq)} \to HC_2OH_3O_{2(aq)}$$

## PROCEDURE:

- Each part of the experiment (except number 12) consists of mixing equal volumes of two solutions in a depression of a spot-plate.
- Use about 15-20 drops of samples for each solution.
- Record your observation at the time of mixing.
- Where there is no visible evidence of reaction (no precipitate formed, and no effervescence observed), determine if heat is evolved by immersing the bulb of a



thermometer into the reaction mixture and noting any increase in temperature (exothermic reaction).

- In each case where a reaction has occurred:
  - Complete and balance the overall equation properly indicating each state designation.
  - o Give the appropriate overall ionic equation.
  - Give the appropriate net ionic equation.
- Where there is **no evidence of reaction**, write the words "No Reaction" as the right hand side of the equation.
- 1. Mix sodium chloride (0.1 M) and potassium nitrate (0.1 M) solutions.
- 2. Mix sodium chloride (0.1 M) and silver nitrate (0.2 M) solutions.
- 3. Mix sodium carbonate 0.1 M and dilute HCl (6 M) solutions.
- 4. Mix 10% sodium hydroxide and dilute hydrochloric acid (6 M) solutions.
- 5. Mix barium chloride (0.1 M) and dilute sulfuric acid (3 M) solutions.
- 6. Mix dilute ammonium hydroxide (6 M) and dilute H<sub>2</sub>SO<sub>4</sub> (3 M) solutions.
- 7. Mix copper (II) sulfate (0.1 M) and zinc nitrate (0.1 M) solutions.
- 8. Mix sodium carbonate (0.1 M) and calcium chloride (0.1 M) solutions.
- 9. Mix copper (II) sulfate (0.1 M) and ammonium chloride (0.1 M) solutions.
- 10. Mix 10% sodium hydroxide and dilute nitric acid (3 M) solutions.
- 11. Mix iron (III) chloride (0.1 M) and ammonium hydroxide (6 M) solutions.
- 12. **DO THIS IN THE HOOD**: Place a few crystals of solid sodium sulfite into the depression of a spot-plate. Add drop wise about 20 drops of dilute hydrochloric acid solution (6 M), and observe.

## Experiment 6: Net Ionic Equations REPORT FORM

| NAME: |    | Date:   | Partner(s): |
|-------|----|---|-------------|
| 1.    |    | ium chloride and potassium nitrate<br>Evidence of reaction  |             |
|       | b. | Overall Equation  |             |
|       | C. | Ionic Equation  |             |
|       | d. | Net Ionic Equation  |             |
| 2.    |    | ium chloride and silver nitrate<br>Evidence of reaction     |             |
|       | b. | Overall Equation  |             |
|       | C. | Ionic Equation  |             |
|       | d. | Net Ionic Equation  |             |
| 3.    |    | ium carbonate and hydrochloric acid<br>Evidence of reaction |             |
|       | b. | Overall Equation  |             |
|       | C. | Ionic Equation  |             |
|       | d. | Net Ionic Equation  |             |

| 4. |    | sodium hydroxide and hydrochloric acid Evidence of reaction  |
|----|----|--|
|    | b. | Overall Equation   |
|    | C. | Ionic Equation   |
|    | d. | Net Ionic Equation   |
| 5. |    | um chloride and sulfuric acid<br>Evidence of reaction        |
|    | b. | Overall Equation   |
|    | C. | Ionic Equation   |
|    | d. | Net Ionic Equation   |
| 6. |    | te ammonium hydroxide and sulfuric acid Evidence of reaction |
|    | b. | Overall Equation   |
|    | C. | Ionic Equation   |
|    | d. | Net Ionic Equation   |
|    |    |  |
|    |    |  |

| 7. |    | per (II) sulfate and zinc nitrate Evidence of reaction         |
|----|----|--|
|    | b. | Overall Equation   |
|    | C. | Ionic Equation   |
|    | d. | Net Ionic Equation   |
| 8. |    | um carbonate and calcium chloride<br>Evidence of reaction      |
|    | b. | Overall Equation   |
|    | C. | Ionic Equation   |
|    | d. | Net Ionic Equation   |
| 9. |    | per (II) sulfate and ammonium chloride<br>Evidence of reaction |
|    | b. | Overall Equation   |
|    | C. | Ionic Equation   |
|    | d. | Net Ionic Equation   |
|    |    |  |

| 10. | 10%<br>a. | sodium hydroxide and nitric acid Evidence of reaction      |
|-----|-----------|--|
|     | b.        | Overall Equation   |
|     | C.        | Ionic Equation   |
|     | d.        | Net Ionic Equation   |
| 11. |           | (III) chloride and ammonium hydroxide Evidence of reaction |
|     | b.        | Overall Equation   |
|     | C.        | Ionic Equation   |
|     | d.        | Net Ionic Equation   |
| 12. | Sod<br>a. | ium sulfite and hydrochloric acid<br>Evidence of reaction  |
|     | b.        | Overall Equation   |
|     | C.        | Ionic Equation   |
|     | d.        | Net Ionic Equation   |
|     |           |  |

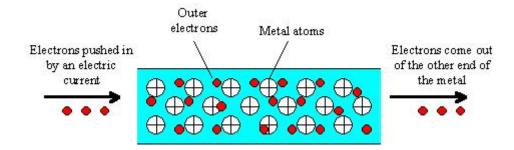
## **EXPERIMENT 7: Conductance in Solutions**

## **PURPOSE:**

- 1. To investigate the phenomenon of solution conductance
- 2. To distinguish between compounds that form conducting solutions from compounds that form non-conducting solutions.

## PRINCIPLES:

Electrical current is a flow of electrons between two points at different potential energy. In solid state, electrical **conductors**, such as metals, the electrons flow through the metal.

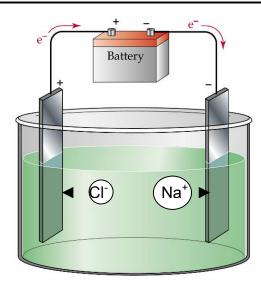


In many solids and liquids, electrons are held too tightly to flow and no measurable electrical conductance is observed. These substances are called **insulators**.

When a solid ionic substance such as sodium chloride, NaCl<sub>(s)</sub>, is tested for conductance we find that it is an insulator, but when molten sodium chloride, NaCl<sub>(l)</sub>, is tested we find that it is a conductor.

Both solid and molten NaCl are made up of Na<sup>+</sup> and Cl<sup>-</sup> ions, but in the molten phase conductance can take place because the ions are mobile (free to move).

Thus electrical conductance can occur by motion of charged species. Negative ions are attracted toward a positive electrode and positive ions are attracted toward a negative electrode.

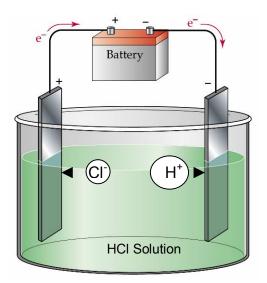


When some solid, liquid or gaseous substance dissolves in a solvent, the substance may produce mobile ions and the resulting solution can conduct electricity in a manner similar to molten  $NaCl_{(i)}$ .

If a substance, such as HCI dissolves in a solvent such as water and separates completely into the component ions (complete ionization), according to the equation given below, it will conduct current well.

$$HCl_{(g)} + H_2O_{(l)} \to HCl_{(aq)} \to H^+_{(aq)} + Cl^-_{(aq)}$$

Such substances are called strong electrolytes.



The observed conductance of a solution of a strong electrolyte will be directly related to the concentration of the electrolyte solution.

Other substances only partially separate into ions (partial lonization), thus giving solutions relatively low conductance. These substances are called **weak electrolytes**.

For example formic acid, HCOOH, ionizes into  $H^+_{(aq)}$  and HCOO $^-_{(aq)}$  to the extent of only one molecule in every one-hundred in a 1M HCOOH aqueous solution, according to the equation given below:

$$100\;HCOOH_{(l)} + X\;H_2O \rightarrow 99\;HCOOH_{(aq)} + 1\;H^+_{(aq)} + 1\;HCOO^-_{(aq)}$$

Some substances dissolve in a given solvent but do not separate into ions at all and their solutions do not conduct electricity. These substances are called **non-electrolytes**.

$$C_{12}H_{22}O_{11(s)} + H_2O \rightarrow C_{12}H_{22}O_{11(aq)}$$

If we write formulas representing **strong electrolytes** in aqueous solution we write them in the **ionic form**.

Formulas of weak and non-electrolytes are written in their molecular form.

## PROCEDURE:

The apparatus is a conductivity indicator (shown below) that consists of a LIGHT EMITTING DIODE (LED) connected to two copper probes (electrodes)



#### SETTING UP THE CONDUCTIVITY INDICATOR

The conductivity indicator should be treated with care and concern.

A 9 volt D.C. power supply is required for operation. The power is available at your bench in the form of a raised electrical panel.

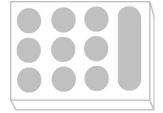
- 1. Connect the two posts of the conductivity indicator to the low voltage power supply with two cords provided.
  - a. The alligator end of the **red cord** is snapped onto the **(+) post** and the other end is inserted into the **red outlet** of the low voltage pane.
  - b. The alligator end of the **black cord** is snapped onto the **(-) post** and the other end is inserted into the **black outlet** of the low voltage panel.
- 2. Test the circuit by touching the tips of the two copper electrodes to a coin or a piece of metal.
  - a. If the conductivity indicator is working properly, the LED will rapidly flash red.
  - b. If no flashing occurs, notify your laboratory instructor.

The response of the LED when both copper electrodes are directly in contact with a solid material or when immersed in a liquid will indicate the following types of conductivity of electric current:

- a. Strong Conductor -----LED will Glow red and blink rapidly
- b. Weak Conductor-----LED will have a dull red glow (no blinking)
- c. Non-Conductor-----LED will not light up

## **USING THE CONDUCTIVITY INDICATOR**

- 1. Clean and dry your Chemplate.
- Fill the depressions in the Chemplate with the liquids to be tested
  - a. Record the name of the liquid with the number of the depression.



- b. Fill the large oval depression in the Chemplate with deionized water.
- 3. Test the substances by using the conductivity indicator
  - a. The position of the conductivity indicator should be perfectly vertical perpendicular to the Chemplate to obtain consistent observations.
  - b. The depth of the two electrodes should always immersed at the same depth to obtain consistent observations.
  - c. Observe and record the reaction of the LED.
  - d. Before testing the next substance, rinse the two probes by immersing them in deionized water which from the large oval depression.

NOTE: The electrodes are clean if the LED does not light up when the electrodes are immersed in D.I. water.

- e. Blot the excess water from the probes with tissue.
- f. Continue to the next depression and repeat the procedure.

NOTE: It is important that the section between the contacts of the probes on the circuit board be kept free of corrosion. This can be accomplished by rinsing and blotting dry the probes after each use. If build-up does occur, notify your instructor as this may cause a short in the circuit.

## **GENERAL INSTRUCTIONS**

- 1. There are 11 stations set-up in the lab
  - a. PART I: Pure substances
  - b. PARTII: Aqueous solutions
  - c. PART IIIA: Factors affecting conductance: Concentration
  - d. PART IIIB: Factors affecting conductance: Extent of ionization
  - e. PART IIIC: Factors affecting conductance: Solvent effects
  - f. PART IVA: Reacting systems: HC<sub>2</sub>H<sub>3</sub>O<sub>2(1)</sub>/H<sub>2</sub>O<sub>(1)</sub>

- g. PART IVB: Reacting systems: HC<sub>2</sub>H<sub>3</sub>O<sub>2(aq)</sub>/NH<sub>3(aq)</sub>
- h. PART IVC: Reacting systems: Ba(OH)<sub>2(aq)</sub>/H<sub>2</sub>SO<sub>4(aq)</sub>

#### NOTES:

- You may start working at any station and may go from station to station in any order depending on availability.
- As you move from station to station
  - DO take with you your Chemplate
  - **DO** rinse and dry your Chemplate between stations
  - **DO** take your stirring rod
  - **DO** rinse the electrodes with D.I. water
  - DO dry the electrodes with tissue
  - DO NOT remove reagents from stations
  - DO NOT leave reagent bottles open
  - **DO NOT** switch the droppers from the bottles
  - **DO NOT** remove or disconnect the conductivity indicator
- 2. For all the tests in PARTI, PART II, and PART III add drop wise 30 drops of the test substance into a depression of your Chemplate. For PART IV, specific instructions are given in that section.
- Observe and record the CONDUCTANCE of each substance using the following keys:
  - + → Strong Conductor → Strong Electrolyte (SE)
  - +/- → Weak Conductor → Weak Electrolyte (WE)
    - → Non-Conductor → Non-Electrolyte (NE)
- 4. If the solute is a Weak Electrolyte or a Non-Electrolyte the predominant solute species are molecules. The formula of the solute is written in Molecular Form (AB).
- 5. If the solute is a Strong Electrolyte the predominant solute species are ions. The formula of the solute is written in Ionic Form (A<sup>+</sup> and B<sup>-</sup>).

## PART I: PURE SUBSTANCES

**NOTE:** For the tests that follow the electrodes must be dry!

Test the conductance of the following solutions and record your results.

1. Glacial acetic acid (HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>, 100% pure)



**CAUTION: DO NOT INHALE THE GLACIAL ACETIC ACID!** 



- 2. Ligroin (a hydrocarbon)
- 3. Deionized water

## PART II: AQUEOUS SOLUT IONS:

**NOTE:** For the tests that follow the electrodes must be well rinsed between tests with D.I. water. But they do not need to be dry.

Test the conductance of the following solutions and record your results.

- 1. Tap water
- 2. 95% ethanol, C<sub>2</sub>H<sub>5</sub>OH<sub>(aq)</sub>
- 3. Sucrose,  $C_{12}H_{22}O_{11(aq)}$ , 1 M
- 4. Acetic acid, HC<sub>2</sub>H<sub>3</sub>O<sub>2(aq)</sub>, 1 M
- 5. Ammonia, NH<sub>3(aq)</sub>, 1 M
- 6. Nitric acid, HNO<sub>3(aq)</sub>,1M
- 7. Ammonium acetate, NH<sub>4</sub>C<sub>2</sub>H<sub>3</sub>O<sub>2(aq)</sub>, 1 M

## PART III: FACTORS AFFECTING CONDUCTANCE:

#### A. Concentration

Test the conductance of the following solutions and record your results.

NOTE: For the tests that follow the electrodes must be very well rinsed between tests with D.I. water, but they do not need to be dry. If the LED comes on when the electrodes are immersed in the D.I. water placed in the Chemplate, you may rinse the electrodes in fresh D.I. water placed in a beaker.

- 1. 1.0 M HCI
- 2. 0.1 M HCI
- 3. 0.01 M HCI
- 4. 0.001 M HCI
- 5. 0.0001 M HCI

#### B. Extent of ionization

Test the conductance of the following solutions and record your results.

- 1. 0.1M NaCl
- 2. 0.1M H2SO4
- 3. 0.1M HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>

#### C. Solvent Effects

Test the conductance of the following solutions and record your results.

NOTE: The electrodes must be dry!

1. Hydrogen chloride dissolved in ligroin



**CAUTION: DO NOT INHALE** 



2. Hydrogen chloride dissolved in water (1.0 M solution)

## PART IV: REACTING SYSTEMS

## A. Pure Acetic Acid / Deionized Water

- a. Place 30 drops of deionized water in one of the depressions.
  - i. Test and record the conductance.



# DRY THE ELECTRODES VERY WELL BEFORE PROCEEDING TO THE NEXT TEST!



b. Place 30 drops of glacial acetic acid (HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub> 100% pure) into another depression.



## **CAUTION! DO NOT INHALE THE GLACIAL ACETIC ACID!**



i. Test and record the conductance



## RINSE AND DRY THE ELECTRODES VERY WELL BEFORE PROCEEDING TO THE NEXT TEST!



c. Place 15 drops of glacial acetic acid (HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>. 100% pure) into a third depression.



## **CAUTION! DO NOT INHALE THE GLACIAL ACETIC ACID!**



- i. Add 15 drops of deionized water to the glacial acetic acid.
- ii. Mix the two liquids in the depression well by using a stirring rod.
- iii. Test and record the conductance of the mixture.
- d. In your report write a balanced equation for the reaction between pure acetic acid and deionized water including state designations.

## B. Acetic Acid Solution/Ammonia Solution

- a. Place 30 drops of 1 M acetic acid solution in one of the depressions.
  - i. Test and record the conductance.



## PROCEEDING TO THE NEXT TEST!



- b. Place 30 drops of 1 M ammonia solution into a depression.
  - i. Test and record the conductance.



# RINSE THE ELECTRODES VERY WELL BEFORE PROCEEDING TO THE NEXT TEST!



- c. Place 15 drops of 1 M acetic acid solution into a depression.
  - i. Add 1 drop of phenolphthalein to the acetic acid solution.
  - ii. While stirring with a rod, add 1M ammonia solution drop wise to the acetic acid solution containing the phenolphthalein indicator until the solution turns and remains faint pink.
  - iii. Test and record the conductance of the pink solution.
- d. In your report write a balanced equation for the reaction between an aqueous solution of acetic acid and an aqueous solution of ammonia including state designations.

## C. Sulfuric Acid Solution / Barium Hydroxide Solution

- Place 30 drops of freshly prepared 0.05 M barium hydroxide solution in one of the depressions.
  - i. Test and record the conductance.



# PROCEEDING TO THE NEXT TEST!



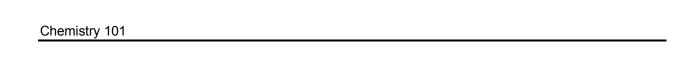
- b. Place 30 drops of 0.05 M sulfuric acid solution into a depression.
  - i. Test and record the conductance.



# RINSE THE ELECTRODES VERY WELL BEFORE PROCEEDING TO THE NEXT TEST!



- c. Place 15 drops of freshly prepared 0.05 M barium hydroxide solution into a depression.
  - i. Add 1 drop of phenolphthalein to the barium hydroxide solution. (The solution should turn pink.)
  - ii. While stirring with a rod add 0.05 M sulfuric acid solution drop wise to the barium hydroxide solution containing the phenolphthalein indicator until the pink indicator color disappears.
  - iii. To check that you did not add too much sulfuric acid, add one drop of barium hydroxide and mix. The mixture should turn pink. If it does not turn pink, keep adding barium hydroxide drop wise until it does turn pink.
  - iv. Now make the mixture turn colorless again by adding drop wise sulfuric acid while mixing well.
    - 1. Repeat these alternate additions of barium hydroxide and sulfuric acid until you are able to make the mixture turn
      - a. Red upon addition of only one drop of barium hydroxide
      - b. Colorless, upon the addition of only one drop of sulfuric acid
  - v. Test and record the conductance of the **colorless** mixture obtained by the addition of only one drop of sulfuric acid.
- d. In your report, write a balanced equation for the reaction between an aqueous solution of barium hydroxide and an aqueous solution of sulfuric acid including the state designations.



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## Experiment 7: Conductance in Solutions REPORT FORM

| NAME: |   | Date                        | e: Partner(s):_                         |  |  |  |  |
|-------|---|-----------------------------|---|--|--|--|--|
| Ρ     | Part I – Pure Substances                  |                             |   |  |  |  |  |
|       |   | Conductance<br>(+, ±, or -) | Electrolyte Character<br>(SE, WE or NE) | Formula of Solute<br>(Predominant Species) |  |  |  |
|       | Glacial Acetic Acid                       |                             |   |  |  |  |  |
|       | Ligroin (C <sub>7</sub> H <sub>16</sub> ) |                             |   |  |  |  |  |
|       | Deionized Water                           |                             |   |  |  |  |  |

## Part II - Aqueous Solutions

|                     | Conductance<br>(+, ±, or -) | Electrolyte Character (SE, WE or NE) | Formula of Solute (Predominant Species) |
|---------------------|-----------------------------|--------------------------------------|---|
| Tap water           |                             |                                      |   |
| 95% ethanol         |                             |                                      |   |
| Sucrose 1M          |                             |                                      |   |
| Acetic acid 1M      |                             |                                      |   |
| Ammonia 1M          |                             |                                      |   |
| Nitric acid 1M      |                             |                                      |   |
| Ammonium acetate 1M |                             |                                      |   |

## Part III – Factors Affecting Conductance

| A: Concentration | Conductance<br>(+, ±, or -) |
|------------------|-----------------------------|
| 1.0 M HCI        |                             |
| 0.1 M HCI        |                             |
| 0.01 M HCI       |                             |
| 0.001 M HCI      |                             |
| 0.0001 M HCI     |                             |

| B: Extent of Ionization                             | Conductance<br>(+, ±, or -) | Electrolyte Character (SE, WE or NE) | Formula of Solute (Predominant Species) |
|---|-----------------------------|--------------------------------------|---|
| 0.1 M NaCl  |                             |                                      |   |
| 0.1 M H <sub>2</sub> SO <sub>4</sub>                |                             |                                      |   |
| 0.1 M HC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> |                             |                                      |   |

| C: Solvent Effects           | Conductance<br>(+, ±, or -) | Formula |
|------------------------------|-----------------------------|---------|
| Hydrogen Chloride in Ligroin |                             |         |
| Hydrogen Chloride in Water   |                             |         |

## Part IV - Reacting Systems

| A: Pure Acetic Acid /              | Conductance  | Electrolyte Character | Formula of Solute     |
|------------------------------------|--------------|-----------------------|-----------------------|
| Deionized Water                    | (+, ±, or -) | (SE, WE or NE)        | (Predominant Species) |
| Deionized Water                    |              |                       |                       |
| Glacial Acetic Acid<br>(100% pure) |              |                       |                       |
| Acetic Acid and<br>Deionized Water |              |                       |                       |

Equation (Including state designations)

| B: Acetic Acid<br>Solution / Ammonia<br>Solution | Conductance (+, ±, or -) | Electrolyte Character (SE, WE or NE) | Formula of Solute<br>(Predominant Species) |
|--|--------------------------|--------------------------------------|--|
| 1M Acetic Acid                                   |                          |                                      |  |
| 1M Ammonia                                       |                          |                                      |  |
| Acetic Acid and<br>Ammonia                       |                          |                                      |  |

Equation (Including state designations)

| C: Sulfuric Acid<br>Solution / Barium<br>Hydroxide Solution         | Conductance<br>(+, ±, or -) | Electrolyte Character<br>(SE, WE or NE) | Formula of Solute<br>(Predominant Species) |
|---|-----------------------------|---|--|
| 0.05M Ba(OH) <sub>2 (aq)</sub>                                      |                             |   |  |
| 0.05M H <sub>2</sub> SO <sub>4 (aq)</sub>                           |                             |   |  |
| Ba(OH) <sub>2 (aq)</sub> and<br>H <sub>2</sub> SO <sub>4 (aq)</sub> |                             |   |  |

Equation (Including state designations)

## **EXPERIMENT 8: The Activity Series**

## **PURPOSE:**

- To study some typical single replacement reactions
- To develop an activity series for a limited number of elements.

## **PRINCIPLES**:

The chemical reactivity of elements varies over a wide range. Some elements, like sodium and fluorine, are so reactive that they are never found in the free, uncombined state in nature. Other elements, like xenon and platinum, are nearly inert and can be made to react with other elements only under special conditions.

The **reactivity of an element** is related to its tendency to **lose or gain electrons**, which is to be oxidized or reduced.

In principle, it is possible to arrange nearly all the elements into a single series in order of their reactivities. A series of this kind indicates which free elements are capable of replacing other elements from their compounds. Such a list is known as an **activity or electromotive series**.

To illustrate the preparation of an activity series we will experiment with a small group of selected elements and their compounds. A generalized single replacement reaction is represented in the form:

$$A + BC \rightarrow B + AC$$

Element A is the more active element and replaces element B from the compound BC. But if element B is more active than element A, no reaction will occur.

Let us consider two specific examples, using copper and mercury:

## Example 1:

A few drops of mercury metal are added to a solution of copper (II) chloride. In this example no change is observed even after the solution has been standing for a prolonged time, hence, we conclude that there is no reaction.

$$Hg_{(l)} + CuCl_{2(aq)} \rightarrow No \ reaction$$

From this evidence we conclude that mercury will not replace copper in copper compounds, and therefore mercury is a less active metal than copper.

## Example 2:

A strip of metallic copper is immersed in a solution of mercury (II) chloride. In this example the copper strip is soon coated with metallic mercury and the solution becomes pale blue-green (the color of Cu<sup>2+</sup> ions in aqueous solution).

$$Cu_{(s)} + HgCl_{2(aq)} \rightarrow Hg_{(l)} + CuCl_{2(aq)}$$

From this evidence we conclude that copper will replace mercury in mercury compounds. Therefore copper is more reactive than mercury and is before mercury in the activity series.

The single replacement equation given above shows that in terms of oxidation numbers, the chloride ion remains unchanged, but the oxidation number of mercury has changed from 2+ to 0, and the oxidation number of copper has changed from 0 to 2+.

Expressed another way, the actual reaction that occurred was the replacement of a mercury ion (Hg<sup>2+</sup>) by a copper atom (Cu<sup>0</sup>).

This can be expresses more simply in the form of a **total ionic equation**:

$$Cu^{0}_{(s)} + Hg^{2+}_{(aq)} + 2Cl^{-}_{(aq)} \rightarrow Hg^{0}_{(l)} + Cu^{2+}_{(aq)} + 2Cl^{-}_{(aq)}$$

Since the chloride ions do not experience chemical change, they are called **spectator ions** and can be removed from the total ionic equation. This results in a **net ionic equation**:

$$Cu^{0}_{(s)} + Hg^{2+}_{(aq)} \rightarrow Hg^{0}_{(l)} + Cu^{2+}_{(aq)}$$

The net ionic equation shows only those reactants that are actually consumed and those products that are actually formed in the reaction. Total ionic equations are an intermediate step in writing net ionic equations, and for this reason they are often omitted. Since single replacement reactions Involve changes in oxidation number, they are also classified as oxidation-reduction reactions.

## **PROCEDURE:**

## NOTES:

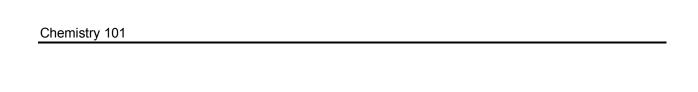
- 1. With some of the combinations used in these experiments the reactions may be slow or difficult to detect. If you see no immediate evidence of reaction, set the tube aside and allow it to stand for about 10 minutes, and then reexamine it.
- 2. Evidence of reaction will be either the evolution of a gas or the appearance of a metallic deposit on the surface of the metal strip. Metals deposited from a solution are often black or grayish (in the case of copper-very dark reddish-brown) and bear little resemblance to commercially prepared metals.

Obtain three pieces of sheet zinc, two of copper and one of lead. Polish the strips (if needed) with fine sandpaper to expose fresh metal surfaces.

Create six test tubes with the following solutions.

| Test Tube | Reactant 1   | Reactant 2                                   |
|-----------|--------------|--|
| 1         | Copper strip | 5 mL 0.2 M AgNO <sub>3</sub>                 |
| 2         | Lead Strip   | 5 mL 0.1 M Cu(NO <sub>3</sub> ) <sub>2</sub> |
| 3         | Zinc Strip   | 5 mL 0.1 M Pb(NO <sub>3</sub> ) <sub>2</sub> |
| 4         | Zinc Strip   | 5 mL 0.1 M MgSO₄                             |
| 5         | Copper Strip | 5 mL 0.1 M H <sub>2</sub> SO <sub>4</sub>    |
| 6         | Zinc Strip   | 5 mL 0.1 M H₂SO₄                             |

Observe the contents of each tube carefully and record any evidence of chemical reaction (gas evolved, metal deposited, change in color of solution).



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## Experiment 8: The Activity Series REPORT FORM

| NAME: | Date: | Partner(s | 3) | :            |
|-------|-------|-----------|----|--------------|
|       | <br>  |           | -, | <del>-</del> |

| Evidence of |   |
|-------------|---|
| reaction    | I. Overall equation                           |
|             | II. Total ionic equation                      |
|             | III. Net ionic equation                       |
| 1           | I. $Cu_{(s)} + AgNO_{3(aq)} \rightarrow$      |
|             | II.   |
|             | III.  |
| 2           | I. $Pb_{(s)} + Cu(NO_3)_{2 (aq)} \rightarrow$ |
|             | II.   |
|             | III.  |
| 3           | I. $Zn_{(s)} + Pb(NO_3)_{2 (aq)} \rightarrow$ |
|             | II.   |
|             | III.  |
| 4           | I. $Zn_{(s)} + MgSO_{4(aq)} \rightarrow$      |
|             | II.   |
|             | III.  |
| 5           | I. $Cu_{(s)} + H_2SO_{4 (aq)} \rightarrow$    |
|             | II.   |
|             | III.  |
| 6           | I. $Zn_{(s)} + H_2SO_{4 (aq)} \rightarrow$    |
|             | II.   |
|             | III.  |
| L           |   |

## QUESTIONS:

1. Complete the following table by writing the symbols of the two elements being compared in each test:

|                  | Tube<br>#1 | Tube<br># 2 | Tube<br>#3 | Tube<br># 4 | Tube<br>#5 | Tube<br>#6 |
|------------------|------------|-------------|------------|-------------|------------|------------|
| Greater Activity |            |             |            |             |            |            |
| Lesser Activity  |            |             |            |             |            |            |

| 2. | Arrange Pb. | . Ma. and | Zn in orde | er of their ac | ctivities, listing | the mo | st active t | first: |
|----|-------------|-----------|------------|----------------|--------------------|--------|-------------|--------|
|    |             |           |            |                |                    |        |             |        |

(1) (2) (3)

3. Arrange Cu, Ag, and Zn in order of their activities, listing the most active first:

(1)\_\_\_\_\_(2)\_\_\_\_(3)\_\_\_\_

4. Arrange Mg, H, and Ag in order of their activities, listing the most active first:

(1)\_\_\_\_(2)\_\_\_(3)\_\_\_\_

5. Arrange all five of the metals (exclude hydrogen) in an activity series, listing the <u>most active first:</u>

(1)\_\_\_\_\_(2)\_\_\_\_(3)\_\_\_\_(4)\_\_\_\_(5)\_\_\_\_

6. On the basis of the reactions observed in the six test tubes, explain <u>why</u> the position of hydrogen cannot be fixed exactly with respect to all of the other elements listed in Question 5?

7. What additional test, or tests, would be needed to establish the exact position of hydrogen among the metals listed in Question 5? Explain how you would interpret the results of the suggested test(s).

- 8. On the basis of the evidence developed in this experiment:
  - a. Would metallic silver react with dilute sulfuric acid? \_\_\_\_\_\_ Why or why not?

b. Would metallic magnesium react with dilute sulfuric acid? \_\_\_\_\_ Why or why not?

9. Three hypothetical metals all form 2+ cations and will be designated as X, Y, and Z. A series of experiments are carried out on these metals and solutions of the ions. The results can be summarized by the equations given below:

$$X_{(s)} + Y^{2+}_{(aq)} \to X^{2+}_{(aq)} + Y_{(s)}$$
  
 $X_{(s)} + Z^{2+}_{(aq)} \to No \ reaction$ 

10. Arrange X, Y, and Z in order of activities, listing the most active first:

(1)\_\_\_\_\_(2)\_\_\_\_(3)\_\_\_\_



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## **EXPERIMENT 9: Standardization of a Base**

## **PURPOSE:**

- To titrate a standard hydrochloric acid solution.
- To determine the exact molarity of a sodium hydroxide solution

## **PRINCIPLES:**

Titration is a convenient volumetric method for accurately determining the concentration of solutions. In general, the process consists of the careful addition of a solution of one reagent whose concentration is to be determined to a solution of another reagent of exactly known concentration until the reaction between these substances is complete. The solution of the reagent of exactly known concentration is called a standard solution and the measured volume of the other solution can be used in calculations to determine the unknown concentration.

A crucial part of a successful titration is that a chemical reaction takes place. However, not all chemical reactions are equally suitable for this type of volumetric analysis. The most successful titrations involve reactions that meet or closely approach the following requirements:

- The reaction should be rapid, so that the titration is not too slow and tedious.
- 2. The reaction should be stoichiometric, i.e., capable of being described by a chemical equation so that the volumetric data can be used directly in the titration calculation.
- 3. The reaction should not involve alternate or side reactions between constituents of the solutions.
- 4. There must be a method for determining when the reaction is complete.

Several general types of reactions come to mind which often meet these criteria. These are:

- 1. Reactions between acids and bases
- 2. Oxidation-reduction reactions
- 3. Reactions involving the formation of complexes
- 4. Precipitation reactions

Only the first of these categories will be considered here.

In general, acid - base titrations are performed by adding controlled quantities of base solution from a buret to a flask containing an accurately known volume of an acid solution. The objective or the titration is to add a quantity or base which reacts completely with an exactly measured volume of acid.

In this experiment the concentration of a basic solution will be determined by measuring the volume of the base required to completely neutralize a precisely measured volume of an acid of known molarity. The acid is hydrochloric acid, HCI, which is an important component in stomach digestive juices. The base is sodium hydroxide, commonly known as lye. The reaction can be written as:

$$HCl_{(aq)} + NaOH_{(aq)} \rightarrow NaCl_{(aq)} + H_2O_{(l)}$$

or more simply:

$$H^{+}_{(aq)} + OH^{-}_{(aq)} \rightarrow H_{2}O_{(l)}$$

Note that one mole of W' ions reacts exactly with one mole of OH-. This implies in turn, that:

#### Number of moles of acid = Number of moles of base

or

(Volume of Acid) x (Molarity of acid) = (Volume of base) x (Molarity of Base)

$$V_{acid} \times M_{acid} = V_{base} \times M_{base}$$

## METHOD:

A solution of sodium hydroxide will be prepared by the dilution of a stock solution of sodium hydroxide. A solution of hydrochloric acid of known concentration will be titrated with a portion of the prepared sodium hydroxide solution using phenolphthalein as an indicator.

From the volume of the sodium hydroxide solution used and the volume and molarity of the hydrochloric acid solution, the molarity of the sodium hydroxide solution will be calculated.

## PROCEDURE:

## 1. Preparation of NaOH solution.

- a. Measure 8.4 mL of 6 M sodium hydroxide solution in a 10-mL graduated cylinder.
- b. Pour the solution into a clean 500-mL plastic bottle.
- c. Dilute this solution to approximately 500 mL with deionized water. (The volume of the sodium hydroxide solution does not have to measured or known accurately, since this solution will be titrated in the next part of the experiment to determine its precise molarity.)

- d. Close the plastic bottle well.
- e. Mix the solution thoroughly by inverting the plastic bottle ten times. (Insufficient mixing of solutions is a common source of error in titrations.)

## 2. Titration of the NaOH solution

- a. Before the titration is begun, all glassware must be thoroughly cleaned. (The glassware usually needed for a titration includes a buret, a pipette, and an Erlenmeyer flask.)
- b. Obtain approximately 125 mL of standard 0.1 M HCl solution in a clean dry 250-mL or 200-mL beaker. (Your beaker must be dry or the water present in the beaker will dilute the hydrochloric acid solution and change the molarity of the solution.)
- c. Record the precise molarity of the hydrochloric acid solution.
- d. Rinse a clean 25-mL volumetric pipette with a 5-mL portion of deionized water.
  - i. Always use a rubber bulb to draw water or solutions into pipette.
  - ii. Make certain that the rinse water contacts the entire inner surface of the pipette.
  - iii. Discard the rinse water.
  - iv. Rinse the pipette at least two times with 5-mL portions of the standard HCl solution by holding the pipette in a horizontal position and rotating it so that the solution contacts the entire inner surface of the pipette.
  - v. Discard the rinse solutions.
- e. Carefully pipette 25.00 mL of the acid solution into the 250-mL Erlenmeyer flask.
  - i. Hold the tip of the pipette against the inner surface of the flask to avoid splatter.
  - ii. When the flow of liquid from the pipette stops, continue to hold the pipette in a vertical position for 15 seconds to allow reproducible draining of the pipette.
  - iii. Touch off the last drop of solution on the tip of the pipette so that the drop enters the flask.
- f. Add about 50 mL of deionized water to the acid solution in the flask to give sufficient volume of solution in which to see a color change.
  - i. The addition of about 50 mL of deionized water to the 25.00 mL of hydrochloric acid will not affect the number of moles of HCl present in the solution, and will not affect the volume of titrant or number of moles of base to be added
- g. Add to the acid solution three drops of phenolphthalein solution and swirl the flask to thoroughly mix the solution.

- h. After the buret has been properly cleaned, rinse the buret with a 5-mL portion of the 0.1 M NaOH solution so that the solution comes in contact with the entire inner surface of the buret and the tip of the buret.
  - i. Drain the rinse solution through the tip of the buret and discard the rinse solution.
  - ii. Repeat this procedure twice with two new 5-mL portions of the NaOH solution.
  - iii. Discard the rinse solution through the tip of the buret.

NOTE: Since the sodium hydroxide solution tends to absorb carbon dioxide from the atmosphere, as shown in the equation below:

$$2NaOH_{(aq)} + CO_{2(g)} \rightarrow Na_2CO_{3(aq)} + H_2O_{(l)}$$

Because of this, *the plastic bottle containing the sodium hydroxide solution should be always closed.* If this precaution is not observed, the concentration of the solution is gradually lowered since the carbon dioxide absorption uses up some of the sodium hydroxide.

- i. Close the stopcock and fill the buret with the solution of base to above the top calibration mark on the buret. After eliminating air bubbles in the tip of the buret, lower the meniscus of the solution until it is at a point on the calibrated portion of the buret.
- j. Record the initial buret reading to the nearest 0.01 mL.
- k. Place your 250 mL Erlenmeyer flask containing the acid solution under the buret and lower the buret tip until it is well into the mouth of the flask as shown.
- Swirl the flask containing the acid solution with the right hand as the stopcock is controlled with the left hand.

#### Notes:

 If the standard HCl solution is approximately 0.1 M and the NaOH solution is also approximately 0.1 M then about 25 mL of NaOH solution should be require for the titration.



- If the concentration of the NaOH is correct, 18 to 20 mL of titrant may be added to the acid solution without danger of adding so much titrant that the end-point of the titration is exceeded.
- As the titration progresses, the approach of the end point will be signaled by a very temporary appearance of the end-point color where the titrant first comes in contact with the acid solution.

- As the end-point is approached more closely, these temporary flashes
  of color persist longer and smaller amounts of titrant should be added.
  Just prior to the end-point, titrant should be added one drop at a time.
  Fractions of a drop may even be added by allowing a droplet to begin
  to form on the buret tip and, after touching the buret tip to the inner
  surface of the flask, by washing down the inner surface of the flask
  with a stream of distilled water from a wash bottle.
- The titrant may splatter slightly as it is added from the buret. If this
  occurs, the inner surface of the Erlenmeyer flask should be washed
  down thoroughly with deionized water before the end-point is reached.
- The titration is complete when the first barely perceptible, but permanent end-point color appears. The best accuracy is obtained if the intensity of the pink color is the faintest that can be seen.
- Divide the last drop of titrant if possible.
- m. Read the final buret volume to the nearest 0.01 mL and record this volume.
- n. Repeat the titration at least two more times.
  - i. Titrations should be done until three results are obtained which agree to within three parts thousand.
  - ii. Between titrations, discard the neutralized solution from the Erlenmeyer flask. Rinse the flask several times with a few milliliters of deionized water.
  - iii. The flask need not be dried before introducing the 25.00 mL of hydrochloric acid for the next titration.
- o. When all of the titrations are completed, drain and rinse the buret thoroughly with deionized water to remove all of the sodium hydroxide solution.
- p. Rinse other glassware also in order to have it ready to use for the next determination.

## CALCULATIONS:

1. For each titration, calculate the molarity of the sodium hydroxide solution.

$$M_{base, moles/L} = \frac{(M_{acid, moles/L})(V_{acid, Liters})}{V_{base, Liters}}$$

Calculate the average molarity of the sodium hydroxide solution.

## 3. Calculate **The Relative Mean Deviation** (RMD) of your base molarity:

$$RMD = \frac{Mean\ Deviation}{Mean\ Molarity}\ x\ 1000 = \frac{\overline{d}}{\overline{X}}\ x\ 1000\ (parts\ per\ thousand)$$

$$\overline{d} = mean \ deviation = \frac{\sum \left| X_i - \overline{X} \right|}{n}$$

 $\overline{X}$  = mean molarity

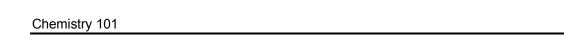
 $X_i$  = individual molarity value

n = number of results

GOOD STUDENT WORK SHOULD HAVE AN RMD OF LESS THAN 3 PARTS PER THOUSAND.

## Experiment 9: Standardization of a Base REPORT FORM

| NAME:  | D            | ate:        | Partner(s    | Partner(s):   |          |  |  |  |  |
|--|--------------|-------------|--------------|---------------|----------|--|--|--|--|
| Molarity of the Standard HCl solution:M        |              |             |              |               |          |  |  |  |  |
| Volume of standard HCl solution used: 25.00 mL |              |             |              |               |          |  |  |  |  |
| Titrations                                     |              |             |              |               |          |  |  |  |  |
| nualions                                       |              |             |              |               |          |  |  |  |  |
|  | First        | Second      | Third        | Forth         | Fifth    |  |  |  |  |
| Final buret reading, mL                        |              |             |              |               |          |  |  |  |  |
| Initial buret reading, mL                      |              |             |              |               |          |  |  |  |  |
| Volume of NaOH, mL                             |              |             |              |               |          |  |  |  |  |
| Molarity of NaOH solution (moles/L)            |              |             |              |               |          |  |  |  |  |
| Average Molarity of NaOH solution: (moles / L) |              |             |              |               |          |  |  |  |  |
|  | First        | Second      | Third        | Forth         | Fifth    |  |  |  |  |
| Deviation  d <sub>i</sub>                      |              |             |              |               |          |  |  |  |  |
| Mean Deviation: (moles / L)                    |              |             |              |               |          |  |  |  |  |
| Relative Mean Deviation, RMD:                  |              |             |              |               |          |  |  |  |  |
| NOTE: Good student wor                         | k should pro | ovide a RMD | of less than | 3 parts per t | housand. |  |  |  |  |



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### **EXPERIMENT 10: Analysis of Vinegar**

### **PURPOSE:**

- To determine the percent by weight of acetic acid in vinegar.
- To perform an Acid-Base titration.

### **PRINCIPLES:**

Vinegar is an aqueous solution of acetic acid ( $HC_2H_3O_2$ ) commonly used in flavoring and preserving food. This is because the acidity adds "tang" and inhibits bacterial growth.

In the investigation that follows the strength of one brand of vinegar will be determined by titration. The acetic acid is neutralized with a standardized sodium hydroxide solution as shown in the following equation:

$$HC_2H_3O_{2(aq)} + NaOH_{(aq)} \rightarrow NaC_2H_3O_{2(aq)} + H_2O_{(l)}$$

Any vinegar sample may be used, but colorless vinegar is preferred because it gives less interference with the observation of the indicator endpoint color change.

As the concentration of the vinegar solution is much higher than the concentration of your standardized sodium hydroxide solution, the original vinegar solution is diluted ten times prior to the titration. This dilution factor must be taken into account when calculating the concentration of the original vinegar solution.

### **PROCEDURE:**

- 1. Clean and dry the following material:
  - a. 500 mL plastic bottle 1
  - b. 100 mL beaker 2
- 2. Clean and rinse the following glassware with distilled water
  - a. 25.00 ml volumetric pipet 6
  - b. 250.0 mL volumetric flask 4 (CAUTION: These are expensive!!)
  - c. 250 mL Erlenmeyer flask 5
  - d. 50 mL Buret 6
- 3. Measure exactly 25.00 mL so of vinegar into a clean 250.0 mL volumetric flask so.

#### Notes:

- Be sure to use a dry beaker to transfer the vinegar.
- Do not put any used vinegar back in the vinegar supply.
- Be sure to rinse your pipette with vinegar prior to using it.
- 4. Dilute the vinegar with deionized water to the mark on the volumetric flask 4.
- 5. Stopper the flask 4 and mix the solution well. (Invert the solution slowly for at least 10 times to completely mix the contents).
- 6. Transfer the dilute vinegar solution to a clean and dry 500 mL plastic bottle and label it with both the contents and your name.
- 7. Immediately wash your volumetric flask 4 with plenty of tap water and several portions of deionized water. Let the flask dry at room temperature.
- 8. Pour about 50 mL of the dilute vinegar solution in your 100 mL beaker 2.
- 9. Rinse your 25.00 mL volumetric pipet **3** several times with portions of diluted vinegar from your beaker.



## Be careful not accidentally add any water to the diluted vinegar solution in the beaker.



- 10. Carefully pipet 25.00 mL **3** of diluted vinegar solution into the 250 mL Erlenmeyer flask **5**.
- 11. Add <u>about</u> 50 mL of deionized water to the Erlenmeyer flask **5**.
- 12. Add 2 drops of phenolphthalein indicator solution and swirl the flask to thoroughly mix the solution.
- 13. Rinse your 50 mL buret **6** several times with a few milliliters of your standardized sodium hydroxide solution.
- 14. Fill the buret **6** with your standardized sodium hydroxide solution.
  - a. Make sure that the tip does not have any air bubbles.
  - b. Record the volume or the buret to the nearest 0.01 mL.
- 15. Titrate the acid sample to a faint pink end point.
- 16. Record the final volume of the buret to the nearest 0.01 mL.
- 17. Repeat the titration procedure described above for at least two more trials. The number of trials run depends on:
  - a. How much standardized sodium hydroxide solution you have available
  - b. The precision of your data.

### **CALCULATIONS:**

Moles of base = (Molarity of base) × (Liters of base)

Moles of acid (diluted vinegar) = Moles of base

Molarity of Diluted Vinegar = 
$$\frac{Moles\ of\ acid\ (diluted\ vinegar)}{Liters\ of\ acid\ (diluted\ vinegar)}$$

The **MOLARITY OF THE ORIGINAL VINEGAR SOLUTION** can be found by keeping in mind that the vinegar has been diluted ten times (from 25.00 mL to 250.0 mL) to obtain the **DILUTED VINEGAR**, whose molarity has been determined by titration with standardized NaOH.

$$\frac{Gramsof \ HC_2H_3O_2}{Gramsof \ vinegar} = \frac{mol \ HC_2H_3O_2}{L \ of \ vinegar} \times \frac{g \ HC_2H_3O_2}{mol \ HC_2H_3O_2} \times \frac{L \ vinegar}{mL \ vinegar} \times \frac{mL \ vinegar}{g \ vinegar}$$

Density of vinegar = 1.0052 g / mL

$$\frac{\% \textit{Weight } HC_2H_3O_2}{\textit{Weight vinegar}} = \frac{\textit{Grams of } HC_2H_3O_2}{\textit{Grams of vinegar}} \times 100$$



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## Experiment 10: Analysis of Vinegar REPORT FORM

| NAME:D                               | Date:                                 | Partner(s): |  |
|--------------------------------------|---------------------------------------|-------------|--|
| Brand of vinegar to be tested:       | · · · · · · · · · · · · · · · · · · · |             |  |
| Molarity of standardized NaOH soluti | on:                                   |             |  |

### **Titrations**

|   | First | Second | Third | Fourth | Fifth |
|---|-------|--------|-------|--------|-------|
| Final buret reading, mL   |       |        |       |        |       |
| Initial buret reading, mL   |       |        |       |        |       |
| Volume of titrant, mL   |       |        |       |        |       |
| Moles of NaOH used  |       |        |       |        |       |
| Moles of HC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> used to<br>neutralize the above number of<br>moles of NaOH |       |        |       |        |       |
| Volume of acid used (L)   |       |        |       |        |       |
| (diluted vinegar)   |       |        |       |        |       |
| Molarity of acid  |       |        |       |        |       |
| (diluted vinegar)   |       |        |       |        |       |
| Mean Molarity of diluted vinegar  |       |        |       |        |       |
| Deviation  d <sub>i</sub>   |       |        |       |        |       |
| Mean Deviation, $\overline{d}$  |       |        |       |        |       |
| Relative Mean Deviation, RMD  |       |        |       |        |       |
| Molarity of Original Vinegar (before dilution)  |       |        |       |        |       |
| $\frac{\textit{Weight of } HC_2H_3O_2}{\textit{Weight of vinegar}}, \%$   |       |        |       |        |       |



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## **EXPERIMENT 11: Stoichiometry Involving a Gas Collected Over Water**

### **PURPOSE**:

 To verify the Ideal Gas Law by solving a stoichiometry problem involving the collection of hydrogen gas over water.

### PRINCIPLES:

The experiment will use a gas eudiometery tube (glass tube used to collect gases over water) to collect and measure a volume of hydrogen gas over water. The hydrogen gas will be generated by reacting magnesium metal with an excess of hydrochloric acid.

By using the Ideal Gas Law, the measurement of volume, pressure and temperature of the hydrogen gas will allow us to calculate the number of moles of hydrogen produced.

$$PV = nRT$$
 Where  $R = 0.08206 \frac{atm \cdot L}{mole \cdot K}$ 

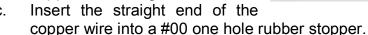
By using stoichiometry the mass of the magnesium that reacted to produce the measured amount of hydrogen can be calculated.

$$Mg_{(s)} + HCl_{(aq)} \rightarrow MgCl_{2(aq)} + H_{2(g)}$$

By comparing this mass of magnesium that reacted (experiment value) with the mass of magnesium actually massed at the beginning of the experiment (theoretical value) we can evaluate the validity of the experimental data and the purity of the magnesium ribbon.

#### PROCEDURE:

- 1. Obtain a piece of magnesium ribbon.
- 2. Mass the magnesium ribbon on the analytical balance.
- 3. Obtain about a four inch piece of copper wire.
- 4. Fold and crumple the magnesium ribbon.
  - a. Wrap the ribbon with the copper wire
  - b. Leave one to two inches of copper wire straight



5. Obtain a 50 mL eudiometer tube.

- 6. Clamp your eudiometer (open side up)
- 7. Add 5 mL of 6M hydrochloric acid.
- 8. Fill the rest of the eudiometer with deionized water to the very top, by pouring slowly from a beaker through a funnel whose stem is touching the inside of the eudiometer (this will minimize premixing of the hydrochloric acid and water, until the tube is inverted for gas collection.)
- 9. Fill a 600 mL beaker with deionized water.
- 10. Sat the copper wire/rubber stopper assembly into the open end of the filled eudiometer and holding your finger over the hole in the stopper invert the eudiometer into a 600 mL beaker containing about 350 mL of deionized water so that the eudiometer is about 2" inches under water.
- 11. Bubbling of hydrogen gas will begin as soon as the hydrochloric acid reaches the magnesium and will continue until all of the magnesium has reacted.
- 12. Any small pieces of magnesium that float to the top and stick to the wall of the wall of the eudiometer should also be reacted by swirling the eudiometer.
- 13. When the bubbling has ceased, wait five additional minutes and then read the volume of the hydrogen gas produced in mL from the graduations of the eudiometer.
- 14. Measure the eight of the water column remaining in the eudiometer that is above the surface of the water in the beaker using a ruler.
- 15. Measure the temperature of the water in the beaker and assume that this is also the same temperature of the aqueous solution in the eudiometer.
- 16. Measure the atmospheric pressure to the nearest 0.1 torr.
- 17. Measure the room temperature.

### **CALCULATIONS**

The sum of the pressure of  $H_2$  inside the tube,  $P(H_2)$ , plus the pressure of the water column remaining in the eudiometer above the surface of the water in the bearer, P(water column) plus the vapor pressure of the water in the eudiometer,  $P(H_2O)$  must equal the atmospheric pressure pressing down on the surface of the water outside the eudiometer. This is only true if all pressure are expressed in the same units.

$$P(atm) = P(H_2) + P(H_2O) + P(water\ column)$$

This relationship can be used to calculate the pressure of hydrogen, P(H<sub>2</sub>), since all other quantities are either measured experimentally or readily available.

Care should be taken to express all pressures in the same units. It is most convenient for the initial calculations of the  $P(H_2)$  to express all other pressures in torr (mm Hg).

The pressure of the water column remaining in the eudiometer, measured in mm  $H_2O$ , must be converted in mm  $H_3$ ; this is done by keeping in mind that the density of water is 1.00 g/mL, whereas the density of mercury is 13.59 g/mL.



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## Experiment 11: Stoichiometry Involving a Gas Collected Over Water REPORT FORM

| NAME:         | Date:   | Partner(s):                | · · · · · · · · · · · · · · · · · · · |
|---------------|---|----------------------------|---------------------------------------|
| DATA:         |   |                            |                                       |
| Mass of mag   | nesium ribbon   |                            | _9                                    |
| Volume of hy  | drogen produced   |                            | _mL                                   |
| Height of wat | er column in eudiometer tube  |                            | _mm H₂O                               |
| Temperature   | of water  | <del></del>                | _°C                                   |
| Temperature   | of air  | <del></del>                | _°C                                   |
| Atmospheric   | Pressure  |                            | _torr                                 |
| CALCULATIO    | <u>ONS:</u>   |                            |                                       |
|               | ate the pressure of hydrogen gas in Equivalent height of water column (d <sub>Hg</sub> = 13.59 g/mL) Show calculations below: |                            |                                       |
| b.            | Vapor pressure of water at the tem (From textbook or handbook)  | perature of the experiment | _torr                                 |
| C.            | Atmospheric pressure  |                            | _torr                                 |
| d.            | Pressure of hydrogen gas<br>Show calculations below:  |                            | _torr                                 |
| e.            | Pressure of hydrogen gas<br>Show calculations below:  |                            | _atm                                  |

| 2. | Calcul | ate the number of moles of hydrogen gas produced   |                |
|----|--------|--|----------------|
|    |        | P (pressure of hydrogen gas)   | _atm           |
|    |        | V (volume of hydrogen gas)   | _L             |
|    |        | T (temperature of air)   | _K             |
|    |        | R (gas constant)   | _atm·L/mole·K  |
|    |        |  |                |
|    |        | N (hydrogen gas)   | _moles         |
| 3. |        | ate the mass of magnesium that has reacted based on experim Write a balanced chemical reaction which represents the remagnesium and hydrochloric acid. Include all state designation | action between |
|    | b.     | Calculate the mass of magnesium that has racted based on moles of hydrogen that have been collected: Show calculations below:  | the number of  |
| 4  | Calcul | Mass of magnesium (experimental value)ate the percent error  | _g             |
|    |        | ·  | a. Ma.         |
|    | a.     | Theoretical value:(The mass of the magnesium used)   | _g Mg          |
|    | b.     | Experimental value:(Calculated from the number of moles of hydrogen collected)   | _g Mg          |
|    | C.     | Percent error:Show calculations below  | _%             |

### **EXPERIMENT 12: Thermochemistry; Heat of Reaction**

### **PURPOSE:**

- 1. To determine the enthalpy change that occurs when aqueous solutions of sodium hydroxide and hydrochloric acid are mixed.
- 2. To determine the enthalpy change that occurs when solid sodium hydroxide is dissolved in water.
- 3. To determine the enthalpy change that occurs when an aqueous solution of hydrochloric acid is mixed with solid sodium hydroxide.
- 4. To determine whether or not your experimental data demonstrates Hess's Law

### **PRINCIPLES:**

Chemical thermodynamics deals with energy changes that accompany physical and chemical transformations. Such changes are usually accompanied by the evolution or absorption of a given amount of heat (q) per mole of product formed.

The observed heat change in a reaction is related to the change in the chemical bonding as the reactants become products.

- If the chemical bonds of the products are more stable than the chemical bonds of the reactants, the system will give off heat to the surroundings (an exothermic reaction).
- If the chemical bonds of the products are less stable than the chemical bonds of the reactants, the system will absorb heat from the surroundings (an endothermic reaction).

The heat change resulting from a reaction carried out at constant pressure is commonly abbreviated  $q_p$ , and is also referred to as enthalpy change ( $\Delta H$ ).

Enthalpy Change = 
$$\Delta H = q_p$$

- For an **exothermic reaction** (heat given off to the surroundings):
  - ΔH is negative
- For an endothermic reaction (heat absorbed from the surroundings):
  - ΔH is positive

The unit of heat commonly used by chemists was a non-SI unit of energy called calorie. The calorie was defined as the amount of heat required to raise the temperature of one gram of water by one degree Celsius.

In 1925 a standard energy unit, the Joule, was introduced. The Joule is presently used as the SI unit of energy.

The calorie and the Joule are related too each other by the following relationship:

$$1 \text{ cal} = 4.184 \text{ J}$$

It follows that 4.184 Joules of heat are required to raise the temperature of one gram of water by one degree Celsius.

The specific heat of any substance(s), is defined as the amount of heat required to raise the temperature of one gram of that substance by 1 °C. By definition the specific of water is therefore 4.184 Joules/g°C.

Specific Heat of Water = 
$$1.00 \frac{cal}{g \cdot {}^{\circ}C} = 4.184 \frac{Joule}{g \cdot {}^{\circ}C}$$

In general, to find the heat, q, required to raise the temperature of a sample of a substance, the following formula is used:

$$q = \Delta H = s \cdot m \cdot \Delta t$$

Where: s = the specific heat of the substance

m = mass of the substance  $\Delta t$  = the temperature change

To measure the heat absorbed or evolved during a physical or a chemical change, a device called a **calorimeter** is commonly used. A calorimeter consists of well-insulated container which gains or loses very little heat to the surroundings.

A calorimeter may be a simple Styrofoam cup covered with cardboard or a small thermos (Snack-Jar). To record the temperature of the calorimeter contents, a thermometer is inserted through the cover of

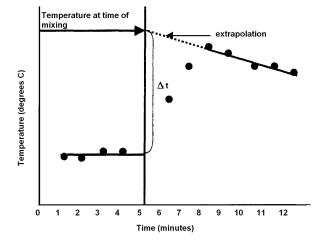
the calorimeter. In this experiment it is assumed that the calorimeter is a perfect insulator, and as such, no heat is exchanged with the calorimeter.



A reaction is usually carried out either in aqueous solution or in contact with a

given mass of water and the respective temperatures measured since waters specific heat is known.

Temperature measurements at the exact time of mixing are rather difficult to obtain since no reliable temperature readings can be taken while the reactants are being mixed. This is why temperature changes are determined by a graphical extrapolation, procedure illustrated in the graph shown.



#### Notes:

- The temperature before the time of mixing (5<sup>th</sup> minute) is typically constant and will be used as the temperature at the time of mixing.
- No temperature reading is taken at the time of mixing (5<sup>th</sup> minute).
- The temperature increases from the 6<sup>th</sup> minute to the 7<sup>th</sup> and then it drops.

The corrected temperature change ( $\Delta t$ ) at the time of mixing is obtained by extrapolating the time-temperature line back to the time of mixing (minute 5). In this experiment the energy change per mole of product formed will be determined for each of the following reactions:

Part II:

$$HCl_{(aq)} + NaOH_{(aq)} \rightarrow NaCl_{(aq)} + H_2O_{(l)}$$
  
1.0  $M$  1.0  $M$  0.5  $M$ 

Part III:

$$NaOH_{(s)} \xrightarrow{H_2O} NaOH_{(aq)}$$

$$0.5 M$$

Part IV:

$$HCl_{(aq)} + NaOH_{(s)} \rightarrow NaCl_{(aq)} + H_2O_{(l)}$$
  
0.5 M

Part V:

Hess's law will be applied to the experimental data obtained.

#### PROCEDURE:

You will be working in <u>pairs</u> and you will require two calorimeters and two thermometers. One calorimeter will be a Styrofoam cup and the second one will be a Snack-Jar.

### PART I: Thermometer Correction Procedure

- 1. Label one thermometer by attaching a small piece of white laboratory tape to the upper end of the thermometer.
- 2. Pour D.I. water (must be at room temperature) into a Styrofoam calorimeter until the cup is at least half full with D.I. water.
- 3. Insert the second thermometer into the D.I. water. Make sure that the mercury bulbs of both thermometers are completely immersed in the water.
- After 1 minute read and record the temperature of each thermometer as carefully as possible to the nearest 0.1 °C. Be careful to avoid parallax in your readings (Read at eye level).

- 5. Determine the temperature correction of the second unlabeled thermometer and record its value. Always use the labeled thermometer in the calorimeter in which the temperature change occurs (the Snack-Jar will be used for this purpose).
- 6. In all subsequent readings apply the necessary correction (the same throughout Part II of the experiment) to the second unlabeled thermometer, so that its readings correspond to that of the labeled thermometer.

### PART II: The Heat of Neutralization of HCl<sub>(aα)</sub> and NaOH<sub>(aα)</sub>: ΔH<sub>2</sub>

1. Set up one calorimeter by placing two Styrofoam cups inside each other and placing the set into a 250 mL beaker for stability. A piece of cardboard with a hole in its center will be used as a lid.



- 2. Your second calorimeter is the Snack-Jar. Set up your second calorimeter by inserting your labeled thermometer through the slit hole of the rubber stopper placed at the center of the Snack-Jar's cover. You may moisten the inside of the rubber stopper to help the thermometer slide easier (Your instructor will demonstrate the safe technique). Find the correct position for the thermometer by sliding it up and down until you are sure than when the cap is screwed on, the mercury bulb of the thermometer is barely touching the bottom of the Snack-Jar.
- 3. Use a graduated cylinder to measure all solutions. Before measuring a solution in a graduated cylinder, be sure the cylinder is clean and has been rinsed with a few mL of the solution to be measured.
  - a. Place 70.0 mL, of 1.00 M NaOH solution into the Styrofoam calorimeter.
  - b. Place and 70.0 mL of 1.00 M HCl solution into the Snack-Jar calorimeter.
- 4. With the lids and thermometers in place, after 1 minute, read and record the temperatures to the nearest 0.1 °C for both calorimeters. Repeat readings at minutes 2, 3, and 4. Apply any necessary corrections to the unlabeled thermometer.
- from the Styrofoam calorimeter into the HCl solution in the Snack-Jar calorimeter. Screw on the cover of the Snack-Jar, making sure that the thermometer is in the correct position. Mix thoroughly the contents of the Snack-Jar with a gently swirling motion (slide it on the bench top) and be prepared to resume taking temperature readings for the 6<sup>th</sup> minute.
- 6. Take temperature readings for the 6<sup>th</sup>, 7<sup>th</sup>, 8<sup>th</sup>, 9<sup>th</sup>, 10<sup>th</sup>, 11<sup>th</sup>, and 12<sup>th</sup> minute.
- 7. Discard the contents of the Snack-Jar calorimeter in the sink. Rinse well both calorimeters with plenty of tap water and a few rinses of D.I. water and drain them well. Wipe the inside of the Snack-Jar calorimeter with soft paper.

- 8. <u>Graph</u> the average temperature of the two solutions before mixing and the temperature of the resulting solution obtained after mixing. In order to assist you in selecting the appropriate scale and format for your graph, a blank graph is attached in the Report Form. (Perform on Day 2)
- 9. Extrapolate the time-temperature line back to the time of mixing (minute 5). (Perform on Day 2)
- 10. Calculate the heat of reaction. Note that the density of the 0.5 M NaCl solution produced after mixing is 1.02 g/mL and its specific heat is 4.06 J/g°C. (Perform on Day 2)
- 11. Calculate the enthalpy of neutralization per mole of water produced. (Perform on Day 2)

### PART III: The Heat of Solution of NaOH(s): ΔH<sub>2</sub>

1. Procure a vial containing about 2.8 g of NaOH pellets from your instructor. Keep the vial tightly closed.





**Note:** NaOH pellets are hydroscopic (they pick up water from the atmosphere). Exposure of the NaOH pellets to air will result in an artificially high mass reading for the pellets.



- 2. Weigh the vial and its contents on the centigram balance (nearest 0.01 g). Keep the vial tightly closed.
- 3. Use a graduated cylinder to transfer 70.0 mL of D.I. water into the Snack-Jar calorimeter. Replace its cover and place the labeled thermometer in position.



- 4. With the cover and thermometer in place on the Snack-Jar calorimeter, as before, read and record the temperature to the nearest 0.1 °C after 1 minute.
- 5. Repeat the temperature readings at minutes 2, 3, and 4.
- 6. At minute 5 do not read the temperature but add all of the  $NaOH_{(s)}$  pellets to the calorimeter and replace the cover and the thermometer.
  - a. Screw on the cover of the Snack-Jar calorimeter, making sure that the thermometer is in the correct position.
  - b. Mix thoroughly the contents of the Snack-Jar calorimeter with a gently swirling motion (slide it on the bench top) and be prepared to resume taking temperature readings for the 6<sup>th</sup> minute. (About 1 -2 minutes of constant stirring will be required for the pellets to completely dissolve).
- 7. As before, read the temperature at minutes 6, 7, 8, 9, 10, 11 and 12.

- 8. Weigh the empty vial and its cap on the centigram balance (nearest 0.01 g) and record its mass. Calculate the mass of the solid NaOH by difference.
- 9. Graph your data as in Part II. Draw the best extrapolation line to minute 5 using data points for minutes 6, 7, 8, 9, 10, 11 and 12. (Perform on Day 2)

NOTE: You may need to ignore data points at minutes 6 and 7 and possibly even 8 since the NaOH pellets probably had not completely dissolved at those times. (Perform on Day 2)

10. Calculate the heat of solution using your  $\Delta t$  value. (Perform on Day 2)

Note that the mass of the solution is about 72.8 g. The specific heat of 1.0 M NaOH is 3.93 J/g°C.

11. Calculate the enthalpy of solution per mole of NaOH<sub>(s)</sub>. (Perform on Day 2)

### PART IV: The Heat of Reaction of HCI(aq) and NaOH(s): ΔH4

1. Procure a vial containing about 2 g of NaOH pellets from your instructor. Keep the vial closed tightly.





**Note:** NaOH pellets are hydroscopic (they pick up water from the atmosphere). Exposure of the NaOH pellets to air will result in an artificially high mass reading for the pellets.



- 2. Weigh the vial and its contents on the centigram balance (nearest 0.01 g). Keep the vial closed tightly.
- 3. Add about 55 mL of 1.0 M HCl solution to a 100 mL graduated cylinder.
- 4. Add D.I. water to the hydrochloric acid solution until you reach the 100.0 mL mark (45 mL of water will be added to the graduated cylinder containing the hydrochloric acid solution).
- 5. Transfer all of the diluted HCl solution to the Snack-Jar calorimeter. Replace the cover and the labeled thermometer in position.
- 6. With the cover and labeled thermometer in place on the Snack-Jar calorimeter, read and record the temperature to the nearest 0.1 °C after 1 minute.
- 7. Repeat the temperature readings at minutes 2, 3, and 4.

- 8. At minute 5 do not read the temperature but add all of the NaOH<sub>(s)</sub> pellets to the calorimeter and replace the cover and the thermometer.
  - a. Screw on the cover of the Snack-Jar, making sure that the thermometer is in the correct position.
  - b. Mix thoroughly the contents of the Snack-Jar with a gently swirling motion (slide it on the bench top) and be prepared to resume taking temperature readings for the 6th minute. (About 1-2 minutes of constant stirring will be required for the pellets to completely dissolve).
- 9. As before, read the temperature at minutes 6, 7, 8, 9, 10, 11 and 12.
- 10. Weigh the empty vial and its cap on the centigram balance (nearest 0.01 g) and record it. Calculate the mass of the solid NaOH by difference.
- 11. Determine Δt for this reaction by extrapolating the data points as in Part III. Again, you may need to ignore data points at minutes 6, 7, and possibly even 8, if not all of the NaOH pellets were dissolved at these times. (Perform on Day 2)
- 12. Calculate the heat of reaction. The density of the resulting solution is 1.02 g/mL and its specific heat is 4.06 J/g°C. (Perform on Day 2)
- 12. Calculate the heat evolved per mole of NaOH on the basis of the mass of NaOH(s) used, as a slight excess of HCl was used to ensure complete reaction of the NaOH<sub>(s)</sub>. (Perform on Day 2)

<u>PART V: This part does not involve experimental work but it is based on the experimental data obtained in Part I, Part II, Part III, and Part IV. (Perform on Day 2)</u>

- 1. Using a sharp pencil (no pen) to plot your data points as exactly as possible.
- 2. Draw straight lines (before and after the time of mixing) that best represent all the data points. Since your experimental measurements are subject to random errors, do not expect the lines to run through all the points.

DO NOT CONNECT CONSECUTIVE POINTS WITH STRAIGHT LINE SEGMENTS!

- As a rule of thumb, there should be about as many experimental points above the line as below.
- The high and low points should be randomly scattered.
- It is wrong to have all the high points to the left and all the low points to the right of the line.
- 3. Extrapolation is the determination of a numerical value of a change that we cannot measure directly (temperature at time 5, time of mixing):
  - is used to draw the straight temperature-time line after minute 5
  - indicates what the temperature of the mixture would have been at the time of mixing (time 5), if at the time of mixing, the heat was instantaneously

and uniformly transferred from the reacting substances to the solution in all parts of the calorimeter.

The temperature at time 5 is determined by EXTRAPOLATION:

A dotted line is drawn (see page 2) which is an extension of the portion of the graph represented by the uniform cooling of the mixture (usually times 8 through 12). The temperature of the mixture at time 5 is the temperature point where the dotted line intersects the vertical line corresponding to time 5.

## Experiment 12: Thermochemistry; Heat of Reaction REPORT FORM

| NAME:                                |                                    | Da   | te:                       | Partner(s):                              |  |
|--------------------------------------|------------------------------------|--|---------------------------|--|--|
| Part I: Thermo                       | meter Correct                      | ion Procedu                                | <u>re</u>                 |  |  |
| Comparison re                        | eadings of the                     | rmometer:                                  |                           |  |  |
|                                      | d thermometer                      |  |                           |  | °C                                     |
|                                      | led thermome                       |  |                           |  | °C                                     |
|                                      | ion to use on                      |  |                           |  | °C                                     |
|                                      | The correction                     |  | •                         |  | 0                                      |
| (Note.                               | ine conection                      | may be pos                                 | sitive of flego           | auve.)                                   |  |
| Part II: The He                      | at of Neutraliz                    | zation of HC                               | l <sub>(ag)</sub> and NaC | $OH_{(aq)}(\Delta H)$                    |  |
| Time (min)                           | Te                                 | mperature (°0<br>NaOH(aq)<br>Styrofoam Cup | C)                        | Temperature (°C)<br>HCI(aq)<br>Snack-Jar | Average Temperature of solution before |
|                                      | Uncorrected                        | eled Thermon<br>Correction                 | Corrected                 | (Labeled Thermometer)                    | mixing (°C)                            |
| 1                                    |                                    |  |                           |  | Plot This                              |
| 2                                    |                                    |  |                           |  | Plot This                              |
| 3                                    |                                    |  |                           |  | Plot This                              |
| 4                                    |                                    |  |                           |  | Plot This                              |
| 5                                    | MIX                                | MIX  | MIX                       | MIX                                      | MIX                                    |
| 6                                    |                                    |  |                           | Plot This                                |  |
| 7                                    |                                    |  |                           | Plot This                                |  |
| 8                                    |                                    |  |                           | Plot This                                |  |
| 9                                    |                                    |  |                           | Plot This                                |  |
| 10                                   |                                    |  |                           | Plot This                                |  |
| 11                                   |                                    |  |                           | Plot This                                |  |
| 12                                   |                                    |  |                           | Plot This                                |  |
| Temperature a                        | at time of mixir<br>raph, by extra | • `  | )                         |  |  |
| Average                              | e Temperatur                       | e of solution                              | s before mix              | king:                                    | °C                                     |
| Temperature of Mixture after mixing: |                                    |  |                           | °C                                       |  |
| Change in temperature (Δt):          |                                    |  |                           |  | °C                                     |

### PART II: Calculations

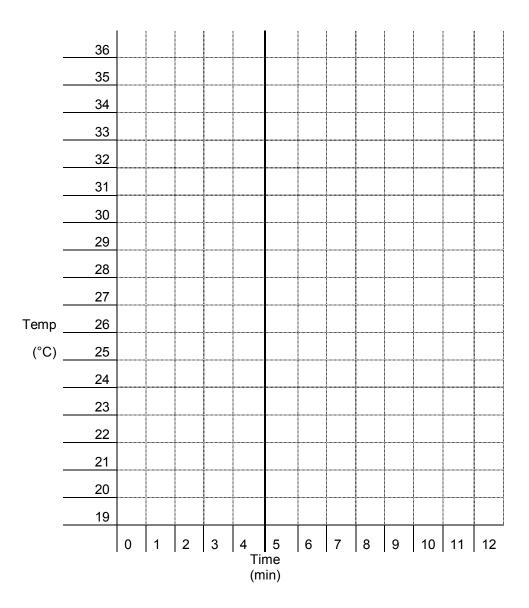
m= g (from 7 above)  $\Delta t = ^{\circ}C$  (from graph)

For the questions and calculations that follow, you must show clearly how you obtained your answers. **NO CREDIT IS EARNED BY UNSUPPORTED ANSWERS.** 

1. Write a balanced chemical equation that illustrates the chemical change taking place in your calorimeter after the mixing of the two solutions. Include state designations. 2. How many moles of HCl have reacted? (HINT: consider the volume and the molarity) 3. How many moles of NaOH have reacted? 4. How many moles of NaCl have been produced? 5. How many moles of H<sub>2</sub>O have been produced? 6. What is the total volume of the mixture? (Ignore the water formed as a product) 7. Calculate the mass of the NaCl solution obtained (d = 1.02 g/mL) 8. Calculate the heat gained by the NaCl solution = total heat of the reaction Express this answer in kJ (Kilojoules) and to the proper amount of significant figures. (The specific heat of the solution is 4.06 J/g°C)  $s= 4.06 J/g^{\circ}C (given)$ 

9. Calculate the enthalpy of neutralization, per mole of  $H_2O$  formed =  $\Delta H_2$  (kJ/mole)

### II. Heat of Neutralization of $HCl_{(aq)}$ and $NaOH_{(aq)}$ , ( $\Delta H_2$ )



### PART III: The Heat of Solution of NaOH<sub>(s)</sub>: ΔH<sub>3</sub>

| Mass of vial + solid NaOH:                     | g  |
|--|----|
| Mass of empty vial:                            | g  |
| Mass of solid NaOH:                            | g  |
| Temperatures at time of mixing (minute 5) (fro |    |
| Temperature of solution:                       | °C |
| ∧t·  | °C |

| Time  | Temperature (°C) |          |
|-------|------------------|----------|
| (min) |                  |          |
|       | water            | solution |
| 1     |                  |          |
| 2     |                  |          |
| 3     |                  |          |
| 4     |                  |          |
| 5     | MIX              | MIX      |
| 6     |                  |          |
| 7     |                  |          |
| 8     |                  |          |
| 9     |                  |          |
| 10    |                  |          |
| 11    |                  |          |
| 12    |                  |          |

### PART III: Calculations

For the questions and calculations that follow, you must show clearly how you obtained your answers. **NO CREDIT IS EARNED BY UNSUPPORTED ANSWERS.** 

1. Write an equation that illustrates the change that takes place in your calorimeter. Include state designations. Note that water is not considered a "true" reactant.

| 2. | Calculate the mass of    | the NaOH solution obtained.  | (Hint: The solution was |
|----|--------------------------|------------------------------|-------------------------|
|    | obtained by adding solid | sodium hydroxide to a measur | ed volume of water)     |

3. Calculate the heat gained by the NaOH solution = Heat of solution Express this answer in kJ (Kilojoules) and to the proper amount of significant figures. (The specific heat of the NaOH solution is 3.93 J/g°C)

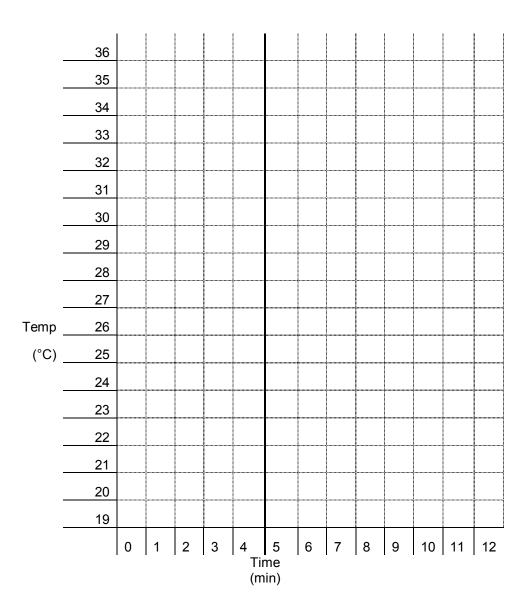
$$s=3.93 \text{ J/g}^{\circ}\text{C}$$
 $m=$  \_\_\_\_\_\_\_ g (from 2 above)

 $\Delta t=$  \_\_\_\_\_\_  $^{\circ}\text{C}$  (from graph)

4. Calculate the number of moles of solid NaOH used:

5. Calculate the enthalpy of solution per mole of NaOH =  $\Delta H_3$  (kJ/mole)

### III. Heat of Solution of NaOH $_{(aq)}$ , ( $\Delta H_3$ )



### PART IV: The Heat of Reaction of HCl<sub>(aq)</sub> and NaOH<sub>(s)</sub>: ΔH<sub>4</sub>

| Mass of vial + solid NaOH:                     | g  |
|--|----|
| Mass of empty vial:                            | g  |
| Mass of solid NaOH:                            | g  |
| Temperatures at time of mixing (minute 5) (fro |    |
| Temperature of solution:                       | °C |
| Λt:  | °C |

| Time<br>(min) | Temperature (°C) |          |
|---------------|------------------|----------|
|               | water            | solution |
| 1             |                  |          |
| 2             |                  |          |
| 3             |                  |          |
| 4             |                  |          |
| 5             | MIX              | MIX      |
| 6             |                  |          |
| 7             |                  |          |
| 8             |                  |          |
| 9             |                  |          |
| 10            |                  |          |
| 11            |                  |          |
| 12            |                  |          |

### PART IV: Calculations

For the questions and calculations that follow, you must show clearly how you obtained your answers. **NO CREDIT IS EARNED BY UNSUPPORTED ANSWERS.** 

1. Write an equation that illustrates the change that takes place in your calorimeter. Include state designations.

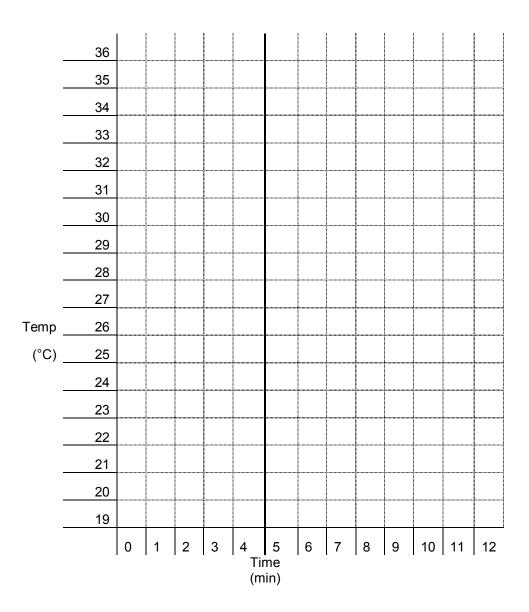
 Calculate the mass of the NaCl solution obtained. (Hint: The volume of the dilute HCl solution is known and it did not change upon the addition of solid NaOH; the density of the resulting NaCl solution is 1.02 g/mL)

3. Calculate the heat gained by the NaCl solution = Heat of reaction Express this answer in kJ (Kilojoules) and to the proper amount of significant figures. (The specific heat of the NaCl solution is 4.06 J/ g°C)

4. Calculate the number of moles of solid NaOH used:

5. Calculate the enthalpy of reaction, per mole of NaOH<sub>(s)</sub> =  $\Delta H_4$  (kJ/mole)

## IV. Heat of Reaction of $HCl_{(aq)}$ and $NaOH_{(aq)}$ , ( $\Delta H_4$ )



### PART V

The reaction of NaOH<sub>(s)</sub> and HCI<sub>(aq)</sub> (PART IV) can be considered to be the algebraic sum of the reaction of NaOH<sub>(s)</sub> and water (PART III) and the reaction of NaOH<sub>(aq)</sub> and HCI<sub>(aq)</sub> (PART II).

Write the molecular equation and the net ionic equation for each reaction.

Include state designations and the enthalpy change for each reaction. Recall that all reactions are exothermic, and as such all enthalpy change values are negative

By analyzing and interpreting your data, indicate whether ot not your data demonstrates Hess' Law.

|                    | Molecular Equation | Net Ionic Equation | ΔH (kJ/mole) |
|--------------------|--------------------|--------------------|--------------|
| Part II            |                    |                    |              |
| Part III           |                    |                    |              |
| Part II + Part III |                    |                    |              |
| Part IV            |                    |                    |              |

| Conclusion: Does your data demonstrate Hess' Law? |  |
|---|--|
| Please explain your answer:                       |  |

# **EXPERIMENT 13: Separation of Cations by Paper Chromatography**

### **PURPOSE:**

To separate and identify cations from a mixture by paper chromatography

### PRINCIPLES:

Substances can be separated by chromatography provided they have different solubilities in two immiscible solvents. The technique involves passing one solvent (the moving phase) through the second solvent (the stationary phase.) Substances that are more soluble in the moving phase will transfer to the moving phase more readily and move along it.

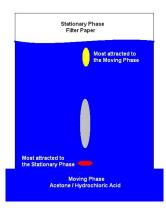
Those substances that are more soluble in the stationary phase will be dissolved in the moving phase more slowly and thus will travel more slowly in the moving phase than the substances that are easily dissolved by the moving phase. This difference in solubility allows substances in a mixture to be separated.

The moving phase may be a gas or a liquid and the stationary phase may be a solid or a liquid absorbed on a solid. This allows chromatographic separations of mixture or gases or liquids and of dissolved solids.

This technique was developed in 1906 by Twsett who separated plant pigment, hence the name chromatography, since his compound were colored. The technique has also been applied to colorless substances, but it is necessary to do something to make them detectable.

We will separate three cations using filter paper as the solid support, filter paper as the stationary solvent and an acetone hydrochloric acid solution as the moving phase. The separation occurs due to the varying affinity of the cations to the solvent as shown in the two pictures below:





### PROCEDURE:



**NOTE:** Perform steps I, II and III as quickly as possibly. Step IV will require about one hour to complete. (You may want to suggest to your instructor to lecture during the elution and not before.)

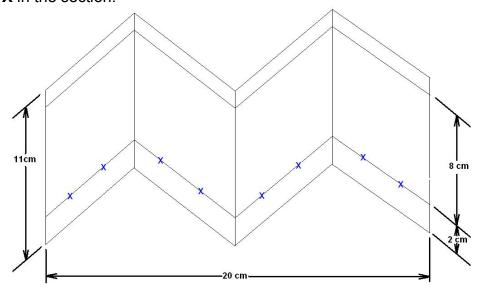


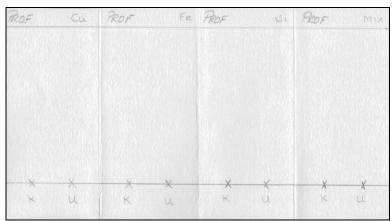
### I. Preparation of the Chromatographic Tank

- 1. Under the fume hood, prepare the eluting solution (moving phase) by carefully pouring 10 mL of 7.2 M HCl and 30 mL of 90% acetone into a 600 mL beaker.
- 2. Pour the liquids down along one side of the beaker to avoid splashes all over the inner walls of the beaker.
- 3. Swirl the liquids gently to mix them. Do not allow the liquids to splash onto the beaker wall.
- 4. Cover the beaker with Plastic-Wrap (seal tightly).
- 5. Find a place for the beaker away from your working area. From this point forward, avoid moving the beaker from this location.
- 6. Allow the beaker and contents to equilibrate.

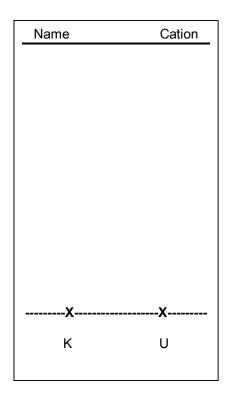
### II. Preparation of the Chromatogram

- 1. Place a rectangular piece of Whatman No. 1 filter paper (11 cm x 20 cm) on a clean dry surface.
- 2. With a pencil (no pen), draw a straight line exactly 2.0 cm from and parallel to one long edge of the chromatography paper.
- 3. Lightly rule a second line 8.0 cm from and parallel to the first line.
- 4. Fold the paper into four equal sections. The folds should be perpendicular to the penciled lines and form accordion type pleats.
- 5. Open the paper with a pencil place two **X**'s in each section on the original line (the 2.0 cm line).
- 6. Each **X** should be 1.5 cm from the edge of the fold and 2.0 cm from the other **X** in the section.





- 7. For each of the four section write the following:
  - a. Top: Your Name
  - b. Top: The cation: Ni<sup>2+</sup>, Cu<sup>2+</sup> or Fe<sup>3+</sup>
  - c. Bottom: K for knownd. Bottom: U for unknown



### Spotting the Standards

- 1. The spotting requires patience and practice. Using a special capillary tube and distilled water, practice getting just the right amount of liquid onto a scrap of Whatman No. 1 paper to produce spots that are between 5-8 mm in diameter.
  - a. Dip one end of the capillary into distilled water (the water will rise in the tube).
  - b. Hold the capillary in the vertical position and touch it lightly to the paper to transfer the liquid.

- c. The size of the spot on the paper is determined by the length of time the capillary touches the paper.
- d. The longer the capillary touches the paper, the more liquid drains out of the capillary and the larger the spot formed on the paper.
- 2. Transfer two (2) drops of each known solution (Fe<sup>3+</sup>, Cu<sup>2+</sup> and Ni<sup>2+</sup>) from the stock bottle to separate (labeled) positions on your spot plate.
- 3. Transfer two (2) drops of the known solution containing all three cations into another separate labeled position on the spot plate.
- 4. Use the liquid in the spot plate to apply the knowns to the proper positions on the chromatographic paper. Make certain that each spot lies on the intersection of its X and the origin line.
- 5. Each spot should be the same size as the practice spots. **Under no circumstances should the spot exceed 0.10 cm in diameter.**

### III. Spotting of Unknown

Note: The unknown may contain one, two, or all three of the possible cations.

- 1. Check out an unknown from the instructor and record the unknown number in your laboratory notebook.
- 2. Remove the label from the vial and stick it onto the space provided for that purpose on your Report Form.
- 3. Write your unknown number on the chromatographic paper.
- 4. Apply the unknown in the four indicated positions.
- 5. Let the spots dry for at least 5 minutes. If a heat lamp is available, place the paper under the heat lamp to speed up the drying process.
- 6. Before going on to the next step, make certain that your spots are completely dry.

#### IV. Elution

- 1. Crease your dried paper on the fold lines to form a "W" shape (when viewed end on.)
- 2. Holding the paper at the top, carefully stand it upright in the beaker containing the elution solution.
  - a. Do not allow the paper to touch the beaker wall.
  - b. Cover the beaker immediately with Plastic Wrap.
- 3. The elution is complete when the solvent front (line of wetness) reaches the top pencil line.
  - a. If the solvent front line does not come evenly to the top pencil line, the elution is considered complete when the solvent front reaches the top pencil line at least at one point.
- 4. When the elution is complete, remove the paper from the beaker.
- 5. Unfold the paper and with a ballpoint pen mark the extreme boundary of the solvent front.
  - a. Mark this quickly, as the solvent evaporates rapidly and completely.
  - b. The solvent front (even or not) must be clearly marked.

6. Place the paper on a clean piece of paper towel and let it dry for a few minutes.

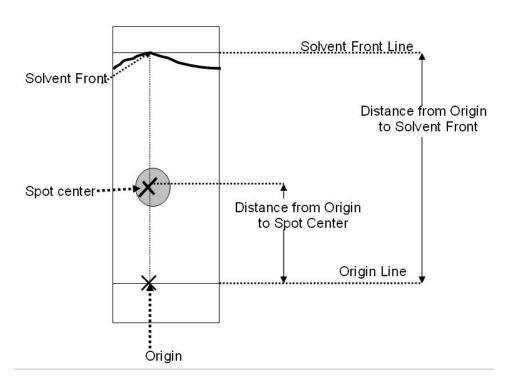
### V. <u>Development of the Chromatogram and Identification of Unknowns</u>

- 1. Cut your chromatogram along the fold lines to separate the four sections.
- 2. Identification of the Cu<sup>2+</sup> ion
  - a. In the hood place the section containing the Cu<sup>2+</sup> ion and the section containing the mixture on the porcelain plate of the large dish containing 15 M ammonia.
  - b. Cover the dish.
  - c. After a few minutes you should notice a blue/green spots forming on the paper.
  - d. Outline the blue/green spots on both papers and mark their centers.
  - e. The blue/green spots indicate the presence of Cu<sup>2+</sup> ions.
- 3. Identification of the Ni<sup>2+</sup> ion
  - a. Treat the section containing the Ni<sup>2+</sup> ion with vapors of ammonia, the same way you did for the Cu<sup>2+</sup> ion.
  - b. At this point you will not see any color that indicates the presence of Ni<sup>2+</sup>.
  - c. Let the paper sit in the ammonia dish for about three minutes.
  - d. Remove the paper from the dish, hold it vertically and spray a solution of 1 % dimethylglyoxime (DMG) solution on the paper.
    - i. The stream of the spray should be directed perpendicular to the paper.
    - ii. Spray the whole section until red spot(s) become visible.
    - iii. Outline the red spots on both papers and mark their centers.
    - iv. The **red spots** indicate the presence of Ni<sup>2+</sup> ions.
  - e. Treat the section containing the mixture with 1% dimethylglyoxime (DMG) in the same manner in which you treated the section containing the known Ni<sup>2+</sup> ions.
- 4. Identification of the Fe3+ ion
  - a. Outline the brown spots on the section containing the Fe<sup>3+</sup> ions and on the section containing the mixture.
  - b. The presence of **yellow spots** indicates the presence of **Fe**<sup>3+</sup> **ions**.
  - c. No chemical development is required to observe these spots.
- 5. The section with the known mixture and the extra unknown spot
  - a. This section was run as an "insurance policy" and should be used to verify the results of the tests run with the other three sections.
- 6. Attach (staple or tape) all four sections of the chromatogram to the Report Form in the spaces provided for this purpose.
  - a. Please place the four sections of the chromatogram side-by-side and not on the top of each other.

### **CALCULATIONS:**

In more complex chromatography, and when characteristic colors are not available, the  $R_f$  (Ratio of Fronts) value helps to identify the chemical species present. The  $R_f$  value is defined by the ratio:

$$R_f = \frac{\text{Distance from Origin to Spot Center}}{\text{Distance from Origin to Solvent Front}}$$



- 1. Use your ruler to measure the required distances to the **nearest 0.1 cm**.
  - a. This is a good enough approximation since the location of the spot centers is not determined very accurately.
- 2. Determine the R<sub>f</sub> values for both the knowns and unknowns.
  - a. Use these values to identify the presence of the ions in your unknown keeping in mind that if two chemical species are identical their  $\mathsf{R}_\mathsf{f}$  values should be reasonably close to each other.

| Chemistry 101 |  |  |  |
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# Experiment 13: Separation of Cations by Paper Chromatography REPORT FORM

| NAME:  |         | Date:                     |           | —<br>Partner(s):           |       |                          |
|--|---------|---------------------------|-----------|----------------------------|-------|--------------------------|
|  |         |                           |           |                            |       |                          |
|  | Spot Ce | om Origin to<br>nter (cm) | Solvent F | om Origin to<br>Front (cm) | (no เ | R <sub>f</sub><br>units) |
| Fe <sup>3+</sup>   | Known   | Unknown                   | Known     | Unknown                    | Known | Unknown                  |
| Cu <sup>2+</sup>   |         |                           |           |                            |       |                          |
| Ni <sup>2+</sup>   |         |                           |           |                            |       |                          |
| Note: If any of the possible cations is not present in your unknown, you will not be able to obtain measurements for that particular cation unknown. If this occurs leave the box(es) blank for that unknown. But all boxes for the knowns must be completed.  Place your chromatograms below: |         |                           |           |                            |       |                          |
| Fe <sup>3+</sup>   |         | Cu <sup>2+</sup>          |           | Ni <sup>2+</sup>           | "     | Insurance<br>Policy"     |
|  |         |                           |           |                            |       |                          |
| CONCLUSIO  | ON:     |                           |           |                            |       |                          |
| Unknown #_   |         | c                         | ontains   |                            |       |                          |

### **EXPERIMENT 14: Atomic Emission**

#### **PURPOSE**:

- To construct an energy level diagram of the hydrogen atom
- To identify an element from its line spectrum.

#### **PRINCIPLES**:

White light, such as emitted by the sun or an incandescent bulb, is a form of energy. When sunlight passes through raindrops it spreads out in a spectrum of colors called **continuous spectrum**. That white light is indeed composed of many colors can also be shown by placing a triangular piece of glass (called a prism) or a diffraction grating in the beam of white light. As the light passes through the prism, the prism changes the direction of some colors more than others. The result is the spreading out of the colors and it is referred to as **dispersion**.

Although we consider light to be colored, color is only a perception in our minds caused by our brains interpreting the sensation of light energy striking the retina of our eyes. There must be some variable property of visible light that tells our eyes and brains what color to see. This property must be different for the various colors of the visible light spectrum.

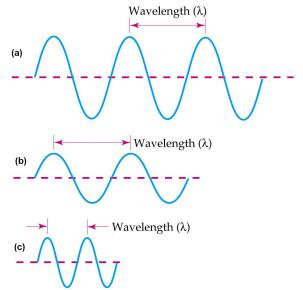
One theory used to describe light considers light as consisting of a series of energy waves that can be transmitted through space. One important characteristic quantity associated with waves is the wavelength, which is the distance from any point on one wave to the corresponding point on the next wave (for example from crest to crest).

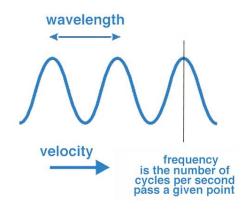
The symbol for wavelength is the Greek letter,  $\lambda$  (pronounced "lambda"). The figure below shows diagrams of three waves, each with a different wavelength. Wave A has the longest wave length and

wave C has the shortest.

Wave length values are expressed in length units, such as meters (m), centimeters (cm), and nanometers (nm) or Angstroms (A).

Note that  $1m = 10^2 cm = 10^9 nm = 10^{10} Å$ 





Another Important characteristic waves is the frequency. The frequency of a wave is the number of waves passing through a point in a interval. The symbol frequency is the Greek letter (pronounced mu).

NOTE:

 $\lambda$  = wavelength = distance from crest to crest

(measured in units of length such as: m, nm, A)

v = frequency = number of waves passing through a point in a

time interval.

(measured in 1/second or Hertz, Hz)

The wavelength multiplied by the frequency is equal to the velocity of the waves.

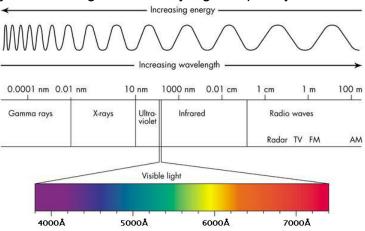
#### WAVELENGTH X FREQUENCY = VELOCITY

This relationship between frequency and wavelength holds true for all types of wave motion. In the case of light traveling through a vacuum, velocity has the specific value of 3.00 X 10<sup>10</sup> cm/sec. This value is an important physical constant and is given the symbol, c. Thus, for all light in a vacuum:

$$c = \lambda \times \nu$$

The various colors of the visible spectrum correspond to various wavelengths of light. Violet light has a relatively short wavelength and red light has a relatively long wave length. Because "c" is a constant for all colors, it is apparent that red light must have a relatively low frequency and violet light a relatively high frequency.

In the spectrum of white light there is a continuous gradation of color; violets gradually merge into blues, blues blend into greens, etc. Because the visible spectrum continuous, it consists of wavelengths from approximately 400 (4000 Å) to 700 nm (7000 Å).



You should realize now that you cannot describe light precisely by just indicating its color. For example, if you wished to specify an orange light, the information in the figure above indicates that the light could have a wavelength around 620 nm (590 nm would be a yellowish orange, while 640 nm would be a reddish orange).

Word descriptions of colors are not exact and vary from person to person. Although catchy phrases such as *scarlet red* and *aqua blue* may serve the world of advertising, scientists need a much more precise system to specify color. This is done by specifying the wavelength (or frequency) range of light that is associated with a particular color.

It is sometimes useful to consider light as consisting of particles rather than waves. This model stresses the concept of light energy and how this energy is converted into other forms. It originated with the physicist, Max Planck, who suggested in 1900 that substances absorb or emit light in discrete amounts called quanta. Albert Einstein extended Planck's theory by proposing that light is composed of particles of energy called photons. Each photon has a definite quantity of energy. When light interacts with a substance, the photon transfers its energy to the substance as a packet. For anyone frequency of light, all the photons have the same energy. The two different models of light, particle and wave, are related quantitatively by the Planck Equation:

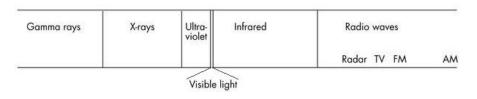
$$E = h \times v$$

NOTE:

E = the energy of the photon h = a proportionality constant called Planck's constant. (h = 6.63 X 10<sup>-34</sup> Joule x second)

All the photons of light of a particular frequency have the same energy. Moreover, as the frequency of light increases, the energy also increases. Therefore, for example photons of blue light have higher energies than photons of red light.

Actually, what we call "light" is only a part of a very broad spectrum of waves called the electromagnetic spectrum. Our eyes see only the part called visible spectrum. Radio and television waves, Infrared rays (IR), Ultra-violet rays (UV), X-rays,  $\gamma$ -rays and cosmic rays are part of the same electromagnetic spectrum, but they cannot be detected by our eyes.



Only a small part of the electromagnetic spectrum is the visible region as shown above. The general regions of the spectrum, such as infrared and ultraviolet, are also indicated. However, there are no sharp boundaries between the parts of the electromagnetic spectrum, just as there are no sharp boundaries within the visible spectrum.

Most substances will emit light energy if heated to a high enough temperature. If the light from an ordinary electric bulb (in which a thin tungsten wire is heated white-hot by an electric current) is passed through a prism, or a diffraction grating a **continuous spectrum**, containing all the wavelengths of visible light is obtained.



Modern street lights produce a different type of light. They consist of high intensity mercury or sodium vapor lamps and are referred to as gas discharge tubes since the element inside the lamp is in its gaseous state and is made to emit light by passing an electric discharge through it. When the spectrum of this light is observed through a prism, only a few bright lines, corresponding to specific wavelengths will be seen.

Each chemical element gives rise to a characteristic **bright line spectrum** (also called atomic emission spectrum) and in a way can be considered as the fingerprint of the element, since no two elements have identical emission spectra. Consequently, the appearance of a line spectrum and the interpretation of the specific spectral lines and their specific wavelengths can be used to identify the element.



The origin of the spectral lines baffled scientists for many years. The explanation of line spectra is based on an intimate knowledge of the atomic structure of the element involved and the energy changes that occur within the atom when energy is absorbed (heat or electrical energy) or released (light energy).

The observation that atoms of a given element which have absorbed energy emit light energy at only fixed wavelengths indicates that atoms can absorb or release energy only in fixed, definite amounts. This in turn implies that an electron in an atom can possess only certain, specific, definite amounts of energy.

This can be summed up by saying that the electron is restricted to specific energy levels in the atoms, usually designated by an integer "n" whose values can be n = 1, 2, 3, 4, 5...  $\infty$ . When n = 1, the electron is in a position closest to the nucleus, and it is in the lowest energy level.

Electrical or heat energy is absorbed when an electron jumps from a lower to a higher energy level (lower to higher value of "n"). The energy thus absorbed is equal to  $\Delta E = E_{final} - E_{initial}$ 

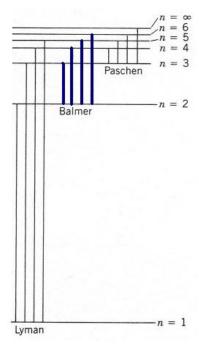
Energy is emitted in the form light energy (brightly colored lines) when the electron "falls" from a higher to a lower energy level (higher to lower value of "n"). The energy thus emitted is also equal to  $\Delta E = E_{\text{final}} - E_{\text{initial}}$ . Summing up the two situations, gives:

#### $\Delta E$ = ENERGY CHANGE (absorbed or emitted) = $E_{final}$ - $E_{initial}$

Niels Bohr, a Danish physicist, studied and explained the origin of the bright lines in the emission spectrum of hydrogen. Hydrogen is the simplest atom (it has only one electron) and hence, it has the simplest atomic spectrum. Four characteristic bright lines can be seen in the visible region of the atomic spectrum of Hydrogen. These lies are called the Balmer lines. The lines result from tile following electron transitions:  $3\rightarrow 2$ ,  $4\rightarrow 2$ ,  $5\rightarrow 2$ ,  $6\rightarrow 2$  (the last one is very difficult to see since it corresponds to a wavelength which is close to the ultra-violet region of the spectrum).

Similar series of lines, which occur in the UV (Lyman series) and IR (Paschen series), have been later identified in the atomic spectrum of hydrogen.

The wavelength of all spectral lines can be calculated by using the equation below:



$$\frac{1}{\lambda} = 109,678 \, cm^{-1} \left( \frac{1}{n_1^2} - \frac{1}{n_2^2} \right)$$

NOTE:

 $n_1$  = final energy level  $n_2$  = initial energy level  $n_2$  >  $n_1$  for emission spectra

Since other elements have a more complex electronic structure, their line spectra are also more complex. However, the pattern of colored lines is characteristic for every element and can be used to identify it.

#### PROCEDURE:

- First, a simplified spectroscope (an instrument that measures light emission) will be calibrated for wavelength measurements by viewing the emission spectrum of helium.
- In the second part of the experiment, the energy level diagram of the hydrogen atom will be determined.
- In the third part of the experiment, the line spectrum of an unknown element will be used to identify the element from a list of possible choices.

#### PART I: CALIBRATION OF THE SPECTROSCOPE

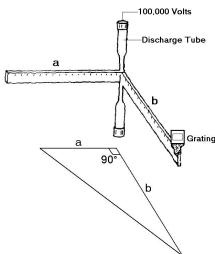
The spectroscope consists of:

- Two meter sticks crossing each other at a right angle
- Diffraction grating

O Gas discharge tube connected to a high voltage power supply. The diffraction grating is a flat piece of plastic with a series of closely spaced lines etched into its surface and it is used instead of a prism to produce the dispersion of light.

The diffraction grating is placed on meter stick "b" in a fixed position exactly 1.00 m away from the point of intersection of the two meter sticks. Meter stick "a" will be used as the scale of the spectroscope and the units marked on meter stick "a" will be used as the arbitrary divisions of the spectroscope.

Calibration is accomplished by viewing the emission spectrum of helium because the emission wave lengths for helium are precisely known.



You will work in pairs and record the positions of the helium spectral lines on the spectroscope scale (meter stick "a"). You will use these data to prepare a "calibration curve" by plotting the measured positions against the known wavelengths of the lines, which are:

| Violet      | 4471 Å                      |
|-------------|-----------------------------|
| Blue        | 4713 Å                      |
| Blue-green  | 4922 Å (not always visible) |
| Light green |                             |
| Yellow      |                             |
| Red         | 6678 Å                      |
| Red         | 7065 Å (not always visible) |

Ask your instructor to position the power supply and helium lamp directly behind the intersection point of the two meter sticks. Check to be certain that the meter sticks are at right angles.



#### **CAUTION**



- 1. The power supply develops several thousand volts. DO NOT TOUCH any portion of the power supply, wire leads, or lamps unless the power supply is unplugged from the wall outlet.
- 2. In addition to visible light, the lamps may emit ULTRAVIOLET RADIATION. Ultraviolet radiation is damaging to your eyes. Use your safety glasses or sunglasses, since they will absorb some of the ultraviolet radiation. DO NOT LOOK DIRECTLY AT ANY OF THE LAMPS WHILE THEY ARE ILLUMINATED FOR ANY EXTENDED PERIOD OF TIME.



**CAUTION** 



- 1. With you instructor's permission, turn on the power supply to illuminate the helium lamp.
- 2. One student will look through the diffraction grating (at eyelash distance) and direct their eye of vision to the left to locate the position of a series of colored lines.
- 3. The second student should move a marker along meter stick "a" until the position of the maker matches the position of the chosen line.
- 4. Record the distance from the point of Intersection of the meter stick to the marker.
- 5. Repeat these steps until you have measured and recorded the distances "a" on the data sheet for all visible lines observed for the helium spectrum.
- 6. Turn off the power supply to the helium lamp.
- 7. On the graph attached to your Report Form plot the measured distances versus the known wavelength for each line.
- 8. Connect the experimental points with a straight line.
  - a. The ideal curve for this relationship is a smooth curve; however for the purposes of this experiment the small deviation from a straight line variation can be considered negligible.
- 9. Attach the graph to your Report Form.



When proceeding from one part of the experiment to the next:

- 1. Do not change the position of the meter sticks
- 2. Do not change the position of the diffraction grating on meter stick "b"



#### PART II: THE ENERGY LEVEL DIAGRAM OF HYDROGEN

- 1. Replace the helium lamp with a hydrogen lamp.
- 2. Repeat the procedure used in PART I to determine the exact positions of the three visible spectral lines of hydrogen.
  - a. You should easily observe the red, blue-green, and violet lines. A fourth line (faint violet) may also be visible, but its position is difficult to determine.
- 3. Record in your laboratory notebook, the color and the location of the hydrogen lines.

4. Use the calibration curve, that you have previously constructed, to determine the wavelengths of the spectral lines of hydrogen and enter these data in Table II.

#### Calculations:

1. Use the following equation to calculate the wavelengths in meters for the four hydrogen lines that appear in the visible region of the spectrum.

$$\frac{1}{\lambda} = 109,678 \, cm^{-1} \left( \frac{1}{n_1^2} - \frac{1}{n_2^2} \right)$$

Remember that  $n_1 = 2$  and that  $n_2 = 3, 4, 5,$  and 6.

- 2. Enter these theoretical (calculated) values in Table III.
- 3. Convert the experimentally determined wavelengths in meters, and enter these values in Table III.
- 4. Calculate the % error for each wavelength.
- Using the same equation, calculate the theoretical values of the wavelengths corresponding to the following transitions: ∞→1, 2→1. Enter: these values in Table IV.
- 6. Complete Table IV with the experimental values of the wavelengths corresponding to the following transitions:  $3\rightarrow2$ ,  $4\rightarrow2$ ,  $5\rightarrow2$ . (Transfer these values from Table III.)
- 7. Using  $c = \lambda \times \nu$ , calculate the frequency corresponding to each wavelength.
- 8. Using  $E = h \times \nu$ , calculate the energy change in Joules for each frequency.
- 9. Convert the energy change values obtained in Joules (J) into Electron volts (eV). Note that  $1 \text{ eV} = 1.60 \text{ X } 10^{-19} \text{ J}$ )
- 10. Using the energy change values expressed in eV ( $\Delta E$ ), calculate the energy values for each energy level ( $E_1$ ,  $E_2$ ,  $E_3$ ,  $E_4$  and  $E_5$ )
- 11. Enter the calculated values in Table V.

HINT: First determine  $E_1$  by keeping in mind that the transition form  $n = \infty$  to n = 1 corresponds to  $\Delta E = E_{\infty} - E_1$  and that  $E_{\infty} = 0$ .

- 12. Construct an energy level diagram of the hydrogen atom on the page provided for this purpose.
- 13. Before you start constructing the diagram, place the energy values you have calculated above on the upper right-hand side corner of the page.

NOTE: Construct the diagram by simply drawing horizontal lines to indicate the energy levels; there is no need to indicate the electron transitions with arrows.

#### PART III: THE IDENTIFICATION OF AN UNKNOWN BY SPECTRAL ANALYSIS

- 1. Repeat the procedure described in PART I, but use as light source a discharge tube containing an unknown element identified only with a number.
- 2. Record the colors and the positions of the spectral lines on meter stick "a" in your laboratory notebook (Table VI).
- 3. Determine the wavelengths of the spectral lines of the unknown element by reading them from the calibration curve and enter these values in your laboratory notebook (Table VI).
- 4. Plot the spectral lines of the unknown element by drawing colored straight vertical lines below the graphical representation of the emission spectra of several elements whose identity is known. (See graph)
- 5. Color all the spectral lines of the elements whose ATOMIC EMISSION SPECTRUM is given. (Refer to the information on the graph which relates the wavelength range to color).
- 6. By matching the position and the color of the spectral lines of your unknown with the position and the color of the spectral lines of the elements given as a possible choice, identify your unknown.

NOTE:Do not expect a perfect match in neither the position nor the number of the spectral lines. Use your judgment to interpret possible errors in wavelength values and missing spectral lines. However, a careful comparison between the color of the spectral lines observed for the unknown and the color of the spectral line of the reference spectra is usually very helpful.



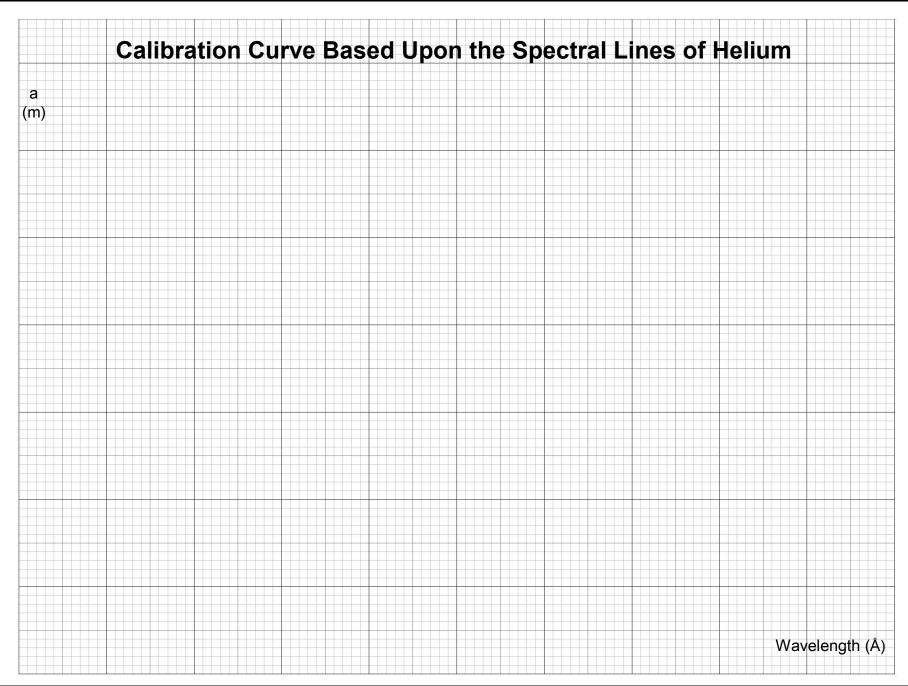
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# EXPERIMENT 14: Atomic Emission REPORT FORM

| NAME: | Date: | Partner(s): |
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### PART I: Calibration of the Spectroscope

| Table I: The Emission Spectrum Of Helium |                             |                |  |  |  |
|--|-----------------------------|----------------|--|--|--|
| Color                                    | Position on meter stick (m) | Wavelength (Å) |  |  |  |
| Violet                                   |                             |                |  |  |  |
| Blue                                     |                             |                |  |  |  |
| Blue – Green                             |                             |                |  |  |  |
| Light Green                              |                             |                |  |  |  |
| Yellow                                   |                             |                |  |  |  |
| Red                                      |                             |                |  |  |  |
| Red                                      |                             |                |  |  |  |



## PART II: The Energy Level Diagram of Hydrogen

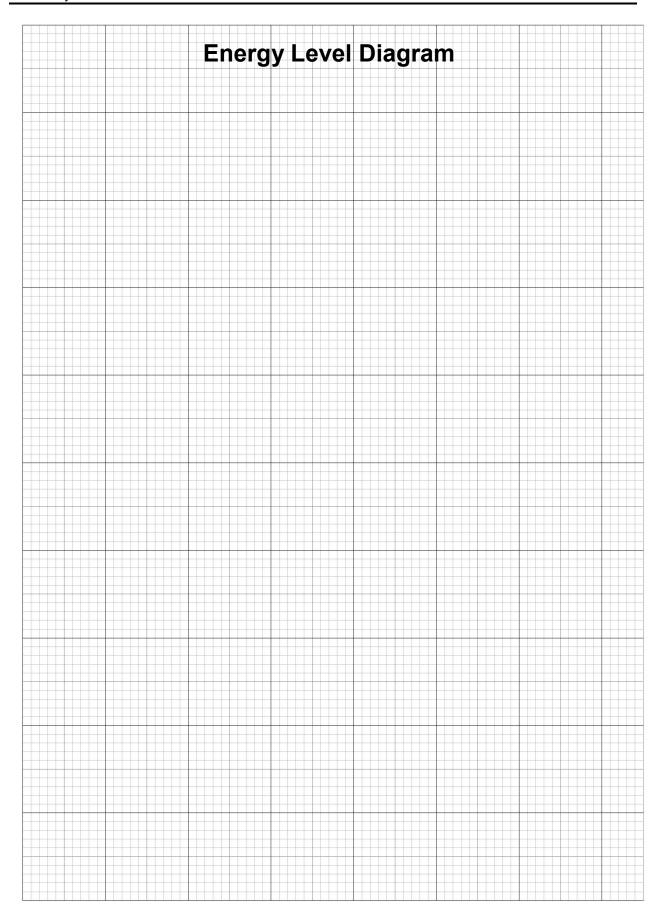
| Table II: Lines Observed in the Emission Spectrum of Hydrogen |                              |                |                  |                            |  |
|---|------------------------------|----------------|------------------|----------------------------|--|
| Color   | Position on meter stick (m)  | Wavelength (Å) |                  | signment<br>on transition) |  |
| Violet  | If not visible, indicate so. |                | n <sub>1</sub> = | n <sub>2</sub> =           |  |
| Blue – Violet   |                              |                | n <sub>1</sub> = | n <sub>2</sub> =           |  |
| Blue – Green  |                              |                | n <sub>1</sub> = | n <sub>2</sub> =           |  |
| Red   |                              |                | n <sub>1</sub> = | n <sub>2</sub> =           |  |

| Table I       | Table III: Experimental versus Theoretical Wavelengths |   |         |  |  |  |
|---------------|--|---|---------|--|--|--|
| Color         | Experimental<br>Wavelength (m)<br>[from Table II]      | Theoretical<br>Wavelength (m)<br>[calculated] | % Error |  |  |  |
| Violet        |  |   |         |  |  |  |
| Blue – Violet |  |   |         |  |  |  |
| Blue – Green  |  |   |         |  |  |  |
| Red           |  |   |         |  |  |  |

$$1m = 10^{10} \text{Å}$$
  $1m = 10^2 \text{cm}$ 

| Table IV: Energy Change Values in the Emission Spectrum of Hydrogen |                              |                            |                                   |           |            |
|---|------------------------------|----------------------------|-----------------------------------|-----------|------------|
| Electron<br>Transition  | Wavel<br>Experimental<br>(m) | ength<br>Calculated<br>(m) | Frequency<br>(sec <sup>-1</sup> ) | ΔE<br>(J) | ΔE<br>(eV) |
| $n_1 = 1, n_2 = \infty$   |                              |                            |                                   |           |            |
| $n_1 = 1, n_2 = 2$  |                              |                            |                                   |           |            |
| $n_1 = 2, n_2 = 3$  |                              |                            |                                   |           |            |
| $n_1 = 2, n_2 = 4$  |                              |                            |                                   |           |            |
| $n_1 = 2, n_2 = 5$  |                              |                            |                                   |           |            |

| Table V: Energy Level Values for Energy Level Diagram |                               |                      |  |  |  |
|---|-------------------------------|----------------------|--|--|--|
| Transition  | Energy Change (∆E) Value (ev) | Energy Value<br>(eV) |  |  |  |
| $\infty 	o 1$   | ΔE =                          | E <sub>1</sub> =     |  |  |  |
| 2 → 1   | ΔE =                          | E <sub>2</sub> =     |  |  |  |
| 3 → 2   | ΔE =                          | E <sub>3</sub> =     |  |  |  |
| 4 → 2   | ΔE =                          | E <sub>4</sub> =     |  |  |  |
| 5 → 2   | ΔE =                          | E <sub>5</sub> =     |  |  |  |



PART III: The Identification of an Unknown by Spectral Analysis

| Table VI: Lines Observed in the Emission Spectrum of Unknown |   |                            |  |  |  |  |
|--|---|----------------------------|--|--|--|--|
| Color  | Position on meter stick (m)   | Wavelength (Å)             |  |  |  |  |
|  |   |                            |  |  |  |  |
|  |   |                            |  |  |  |  |
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|  |   |                            |  |  |  |  |
|  |   |                            |  |  |  |  |
| Unknown #  | is  |                            |  |  |  |  |
| of the spectral lines observe                                | discrepancies between the ned in the spectrum of your uctral lines of the known eleme | inknown versus the number, |  |  |  |  |
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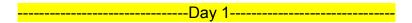
## **EXPERIMENT 15: The preparation and properties of NaHCO<sub>3</sub>**

#### **PURPOSE:**

- The synthesis of NaHCO<sub>3</sub> commonly called:
  - Sodium hydrogen carbonate
  - Sodium bicarbonate
  - Baking soda
- The determination of some of the properties of NaHCO<sub>3</sub>.

#### **BACKGROUND:**

Sodium hydrogen carbonate is an important industrial chemical used in baking, as an antacid, and for the preparation of sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>).



#### PART I: Preparation of Sodium Hydrogen Carbonate

#### Procedure:

- I. Sodium chloride solution preparation
  - (a) Mass approximately 15 g of NaCl on the centigram balance
  - (b) Record the mass to the nearest 0.01 g.
  - (c) Place the NaCl into a 250 mL Erlenmeyer flask.
  - (d) In the hood, add 35.00 mL of D.I. water and 20.0 mL of concentrated ammonium hydroxide solution to the flask.
    - a. Concentrated ammonium is 29% NH<sub>3</sub>, 15.0 M with a density of 0.90 g/mL.

$$NH_{3(g)} + H_2O_{(l)} \Leftrightarrow NH_4OH_{(aq)}$$

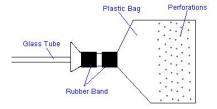


The addition of the NH4OH to the salt water solution should be done under the fume hood.



- b. Stopper the flask after the addition of the concentrated ammonium hydroxide solution.
- (e) Swirl the contents of the flask for about 10 to 20 minutes until all of the sodium chloride dissolves.
- II. Carbon dioxide bubbler adapter preparation
  - (a) Place a small polyethylene bag (1 cm X 5 cm) on a piece of plywood or cardboard.

- (b) Using a small sewing needle secured in a rubber stopper and cardboard as a backing make about 30 perforations completely through the lower 1 cm portion of the plastic bag,
- (c) Make rubber bands by cutting 2 pieces of 2 mm length of rubber tubing (3/16 inside diameter).
- (d) Roll the rubber bands onto a 10 12 inch piece of glass tubing (8 mm outside diameter.)
- (e) Open the perforated plastic bag and place it on the end of the 10 12 inch piece of glass tubing.
- (f) Secure the plastic bag tightly to the end of the glass tubing by rolling the rubber bands onto the top of the plastic bag.



#### III. Carbon dioxide gas generator assembly

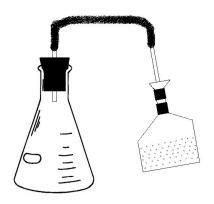
(a) Place approximately 40 g of dry ice lumps, (solid carbon dioxide) about 1/2 inch in diameter, into a 500 mL Erlenmeyer flask.



DO NOT TOUCH THE DRY ICE WITH YOUR HANDS. HANDLE THE DRY ICE WITH A SPOON OR WITH CRUCIBLE TONGS.



- (b) Insert a short length of glass tubing into a #6 one-hole rubber stopper.
- (c) Insert about 14 inches of rubber tubing and connect to the glass tubing in the rubber stopper.
- (d) Connect the bubbler tube prepared in Part II to the flask containing the dry ice as shown in the picture to the right.



#### IV. Apparatus Assembly



#### PERFORM THE FOLLOWING IN THE FUME HOOD



- (a) Secure a 100 mL graduated cylinder to a ring-stand with a clamp.
- (b) Pour the NaCl/NH<sub>3</sub> solution into the graduated cylinder.
- (c) Place the flask containing the ice on several pieces of folded paper towel. This will properly insulate the flask, so that the CO<sub>2</sub> bubbling rate is not too rapid.

- (d) Record the time while placing the CO<sub>2</sub> bubbler tube into the NaCl/NH<sub>3</sub> solution so that the perforated part of the bubbler is at the bottom
- (e) Several streams of bubbles should pass completely through the column of solution in the cylinder. This will ensure the most complete reaction in the shortest possible time.
- (f) Leave the apparatus in the fume hood for at least one half hour

#### V. Sodium hydrogen carbonate formation

(a) After 15 - 20 minutes, a fine suspension of NaHCO<sub>3</sub> particles will form according to the following reactions:

$$\begin{split} CO_{2(g)} + H_2O_{(l)} & \Leftrightarrow H_2CO_{3(aq)} \\ H_2CO_{3(aq)} + NH_4OH_{(aq)} & \to NH_4HCO_{3(aq)} + H_2O_{(l)} \\ NH_4HCO_{3(aq)} + NaCl_{(aq)} & \to NaHCO_{3(aq)} + NH_4Cl_{(aq)} \end{split}$$

- (b) About the same time, the rate of bubbling will slow down. This is an indication that the small holes in the bubbler are starting to plug with solid NaHCO<sub>3</sub>. To prevent the bubbler from becoming completely plugged, remove the bubbler from the graduated cylinder and immerse it for a few seconds in a beaker of hot water to dissolve the NaHCO<sub>3</sub> plugging the holes. **This may have to be repeated several times.**
- (c) Toward the end of the procedure, the plastic bag may be completely removed and the glass tube may be replaced in the solution (bubbles will now come out directly from the glass tube.



Failure to take these precautionary measures may result in the stopper popping out of the flask containing the dry ice, or the bubbler popping out of the graduated cylinder and consequent spillage of solution and solid product.



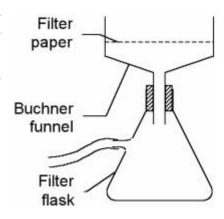
(d) After CO<sub>2</sub> has passed through the solution for a total of 40 minutes, remove the bubbler tube from the solution.

### VI. Reaction mixture filtration

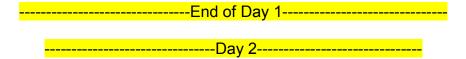
- (a) Assemble a vacuum filtration apparatus using a 500 mL filtration flask and a plastic Buchner funnel (43 mm). See Figure to the right.
- (b) Use a utility clamp and a ring stand to hold the filter flask in place.
- (c) Connect the flask to the vacuum source with vacuum tubing.



- (d) Place two pieces of Whatman # 1 filter paper (42.5 mm) into the Buchner funnel
- (e) Dampen the filter paper with D.I. water and apply the vacuum to secure the paper in place. Turn off the vacuum.
- (f) Swirl the reaction mixture in the graduated cylinder to suspend as much solid as possible of the solution. (The use of a long glass rod will make the mixing easier.



- (g) Rapidly pour about one half of the mixture into the Buchner funnel.
- (h) Turn on the vacuum to remove the filtrate from the solid.
- (i) Again swirl the contents in the graduated cylinder, and rapidly pour the remainder of the mixture into the funnel to remove the filtrate from the solid.
- (j) Turn off the vacuum, remove the Buchner funnel and pour the filtrate into a 250 mL beaker.
- (k) Replace the funnel in the flask and turn on the vacuum.
- (I) Use two 10 15 mL aliquots of the filtrate to rinse any solid NaHCO<sub>3</sub> from the walls of the graduated cylinder.
- (m)Pour each rinse mixture through the Buchner funnel to collect the NaHCO<sub>3</sub>.
- (n) Turn off the vacuum and remove the Buchner funnel from the flask.
- (o) Mass a large watch glass.
- (p) Transfer the precipitate to the large watch glass and place in your locker.



- (q) Determine the mass of dry NaHCO<sub>3</sub>.
- (r) Calculate the percent yield of NaHCO<sub>3</sub> (Note; CO<sub>2</sub> gas is in excess)
- (s) Place your dry product in a plastic bag and label it with the following information:

NaHCO₃ XXX.XX g Your Name

- (t) Seal the bag with tape and staple it to the upper-right hand side of your report.
- (u) Turn In the product along with your lab report to be graded.

#### PART II: PROPERTIES OF SODIUM HYDROGEN CARBONATE

#### Procedure:

- 1. Use a small portion of sodium hydrogen carbonate (NaHCO<sub>3</sub>) (about the size of a pea) and place it into a small test tube.
- 2. Add several drops of 1M HCI.
- 3. Note and record any results in your laboratory notebook.
- 4. Place a small portion of sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) in a test tube.
- 5. Add several drops of 1M HCI.
- 6. Note and record any results in your laboratory notebook.
- 7. Dissolve a small portion of sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) in about 5 mL of D.I. water.
- 8. After the Na<sub>2</sub>CO<sub>3</sub> completely dissolves (as evidenced by a completely clear solution in the test tube), add 1 mL of 1M CaCl<sub>2(ao)</sub> solution.
- 9. Note and record any results in your laboratory notebook.
- 10. To detect the carbon dioxide in your breath, add about 5 mL of clear limewater (aqueous solution of 0.02 Ca(OH)<sub>2(aq)</sub>) to a wide and short test-tube (25 x 150 mm).
- 11. Cover the mouth test tube with a piece of aluminum paper and punch a hole through the foil for a soda straw.
- 12. Gently blow your breath into the straw and through the limewater for several minutes.
- 13. Note and record any results in your laboratory notebook.



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# $\frac{\text{EXPERIMENT 15: The preparation and properties of NaHCO}_3}{\text{REPORT FORM}}$

| NAME:                                      | Date:                 | _ Partner(s): |
|--|-----------------------|---------------|
| PART I: Preparation of Sodiu               | ım Hydrogen Carbonate | <u>9</u>      |
| Mass of NaCI:                              |                       | g             |
| Moles of NaCl:                             |                       | Moles         |
| Show calculations below:                   |                       |               |
| Volume of NH₄OH solution: _                |                       | mL            |
| Molarity of NH <sub>4</sub> OH solution: _ |                       | M             |
| Moles of NH <sub>4</sub> OH                |                       | Moles         |
| Show calculations below:                   |                       |               |
| Which reactant is the LIMITIN              | NG REAGENT?           |               |
| Theoretical yield of NaHCO <sub>3</sub> :  |                       |               |
| Show calculations below:                   |                       |               |
| Mass of watch glass:                       |                       | g             |
| Mass of watch glass+NaHCC                  |                       |               |
| Actual yield of NaHCO <sub>3</sub> :       |                       |               |
| PERCENT YIELD OF NaHCO                     |                       |               |
| Show calculations below:                   |                       |               |

## PART II: PROPERTIES OF SODIUM HYDROGEN CARBONATE

| 1. | What did you observe when 6M HCl was added to NaHCO <sub>3</sub> ?   |
|----|--|
| 2. | Write the balanced chemical equation with state designations for the reaction between NaHCO $_3$ and 6M HCI.           |
| 3. | What did you observe when 6M HC was added to Na <sub>2</sub> CO <sub>3</sub> ?   |
| 4. | Write the balanced chemical equation with state diagrams for the reaction between $\text{Na}_2\text{CO}_3$ and 5M HCI. |
| 5. | What did you observe when the $CaCl_{2(aq)}$ was added to $Na_2CO_3$ ?   |
|    |  |

| - | , -  |
|---|--|
| 6 | . Write the balanced chemical equation with state designations for the reaction between CaCl <sub>2(aq)</sub> was added to Na <sub>2</sub> CO <sub>3</sub> . (Note: Water is neither a reactant nor a product in this reaction.) |
| 7 | . What did you observe when your breath was bubbled through $\text{Ca}(\text{OH})_{2(\text{aq})}$ ?  |
| 8 | . Write a balanced chemical equation with state designations that illustrates the reaction taking place when your breath bubbles through $\text{Ca}(\text{OH})_{2(\text{aq})}$   |
|   |  |



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### **EXPERIMENT 16: The Effect of Temperature on Solubility**

#### **PURPOSE:**

- 1. To determine the solubility of KNO<sub>3</sub> (potassium nitrate) at various temperatures.
- 2. To draw a solubility curve for potassium nitrate.
- 3. To determine the solubility of KNO<sub>3</sub> at 25.0 °C and compare it with the literature data.

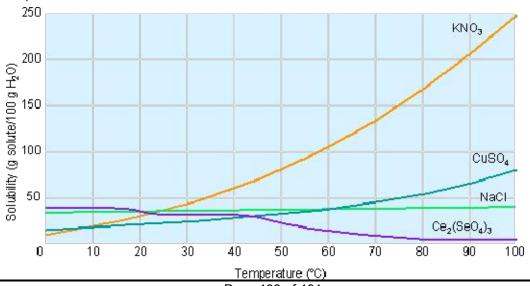
#### PRINCIPLES:

The term **solubility** in chemistry has both general and specific meanings. In everyday situations, we say that a substance is soluble when a sample of that substance can be dissolved in a particular solvent (for example, table salt is soluble in water).

In a more specific sense, the solubility of a substance of a substance refers to a definite numerical quantity. Typically, the solubility of a substance is indicated as the maximum number of grams of the substance that can be dissolved in 100 g of solvent. Since for aqueous solutions the solvent is water, the solubility of these solutions could also be indicated as the maximum number of grams of the substance that can be dissolved in 100 mL of water (recall that the density of water is very near to 1.0 g/mL under most conditions).

Since solubility changes with temperature, handbooks of chemical data that give the solubilities of various substances always indicate the temperature at which the solubility was determined. For most solid substances, the solubility increases with increased temperature. However, for gases and a few solid substances, the solubility decreases if the temperature is raised.

For convenience, to express the dependence of solubility of a particular substance on temperature, a **graph of solubility as a function of temperature** is plotted below.



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#### PROCEDURE:

#### PART I: DETERMINATION OF THE INITIAL SATURATION TEMPERATURE

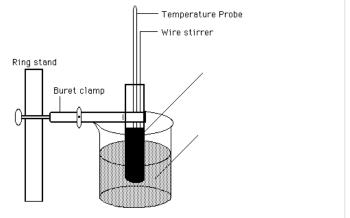
1. Fit a 25 mm x 200 mm test tube with a 2-hole rubber stopper.



Note: It is important that the test tube you use in this procedure be clean and dry. If you test tube is wet, exchange it with a dry one from the stockroom.



- 2. Carefully insert a thermometer through the slit hole of the rubber stopper in such a way that the thermometer bulb is about one-half inch from the bottom of the test tube and that the thermometer can be read from 0°C to 100°C.
- 3. The thermometer slides easily up and down inside the hole, if the inside of the rubber stopper is moistened with 1-2 drops of water.
- 4. Through the other hole in the rubber stopper, insert a metallic stirrer. The metallic stirrer should be long enough to extend from the bottom of the test tube to at least 2 inches above the stopper. By moving this stirrer up and down, a solution in the test tube can be agitated.
- 5. Place about 300 mL of water in a 400-mL beaker, and heat the water to boiling.
- 6. While the water is heating, obtain from the instructor a vial containing about 5 grams of KNO<sub>3</sub> (potassium nitrate).
- 7. Mass and record the mass of the vial and contents to the nearest 0.01 g.
- 8. Open the vial and transfer the contents into your **clean and dry** test tube.
- 9. From a buret dispense 5.00 mL of deionized water into the test tube containing the salt. Record the precise volume of water added.
  - In the following procedure, you must record each time a portion of water is added from the buret. It is essential to know the exact volume of water at each point in the determination.
- 10. Attach the stopper with thermometer and stirrer, and clamp the test tube
  - vertically in the hot water bath (You may stop heating the water at this point).
- 11. Adjust the thermometer so that the bulb of the thermometer will be immersed in the solution in the test tube.
- 12. The test tube should be set up so that the contents of the test tube are immersed fully in the hot water (the liquid levels



inside and outside of the test tube should be about the same).

- 13. Stir the salt in the test tube until the KNO<sub>3</sub> is dissolves.
  - a. If the KNO<sub>3</sub> is not completely dissolved when the temperature of the mixture reaches at least 80<sup>o</sup>C, add exactly 1.00 mL of water from the buret to the mixture.
  - b. Record the exact volume of water added to the test tube.
  - c. Again heat the test tube and contents in the hot water bath to at least 80°C.
  - d. Stir the salt in the test tube until it dissolves.
  - e. Repeat the above procedure, until just enough deionized water has been added to dissolve all of the KNO<sub>3</sub> at a temperature no higher than 80<sup>o</sup>C.



Minimize the amount of time the test tube spends in the hot water to restrict any possible loss of water from the test tube by evaporation.



- 14. After the KNO<sub>3</sub> has completely dissolved, raise the test tube and contents out of the hot water bath and allow the solution in the test tube to cool, while stirring the solution continuously).
- 15. Observe the temperature of the solution carefully, and note the temperature at which the **first crystals of salt begin to form**.
  - a. It may be difficult to read the temperature, since water vapor may condense onto the inside walls of the test tube. The condensation may be removed by wiping the inside walls of the thermometer just in front of your line of vision.
  - b. Make sure you do not overdo the wiping, since that would alter the total volume of solution that you carefully recorded. If you are unsure of the correct reading of the saturation temperature, it is possible to make a second determination, by reheating the solution to dissolve the salt and noting again the temperature at which the first crystals form.

#### PART II: DETERMINATION OF ADDITIONAL SATURATION TEMPERATURES

- 1. Add 1.00 mL of additional deionized water to the test tube. Record.
- 2. Reheat the test tube in boiling water until all the solid has dissolved.
- 3. Remove the test tube from the boiling water and allow it to cool.
- 4. Make a determination of the saturation temperature for the solution in the same manner as indicated earlier. (Repeat, if needed).
- 5. Repeat the addition of 1.00 mL water samples, with determination of the saturation temperatures. When the solution becomes quite dilute and the saturation temperature reaches room temperature, add ice to the water bath to obtain several readings between room temperature and 0°C. In all, about 8 or more saturation temperatures are required in order to obtain a meaningful

solubility curve (2 or 3 of these saturation temperatures should be below room temperature, which is commonly taken to be 25°C)

#### NOTES:

- (a) If the saturation temperature drops sharply upon the addition of the 1.00 mL portions of water, reduce subsequent additions of water to 0.50 mL
- (b) If the saturation temperature does not change enough upon the addition of the 1.00 mL portions of water, increase subsequent additions of water to 2.00 mL.
- (c) In either case, keep accurate records of how much water is added.

#### **RESULTS AND CALCULATIONS:**

1. From your data at each of the saturation temperatures, calculate the solubility of KNO<sub>3</sub>. This is the mass of KNO<sub>3</sub> that would dissolve in 100 g of water at that temperature. Assume that the density of water is exactly 1.00 g/mL, so that your buret additions in milliliters will be equivalent to the mass of water being added.

Example:

If 4.85 g of KNO<sub>3</sub> dissolves in 5.00 g of water (5.00 mL of water):

$$SOLUBILITY = \frac{mass\ of\ KNO_3}{mass\ of\ water}\ x\ 100 = \frac{4.85g\ of\ KNO_3}{5.00g\ of\ water}\ x\ 100 = 97.0 \frac{g\ of\ KNO_3}{100g\ of\ water}$$

- 2. On a piece of graph paper, plot the solubility curve for KNO3, using saturation temperatures on the horizontal axis and solubility's for 100 g of water on the vertical axis. Draw the solubility curve for KNO3 to best fit the experimentally determined points. Some solubility curves are given as an example in Figure Note that all solubility curves are smooth curves and neither straight lines, nor "connect-dots" diagrams. Attach the graph to your report form.
- 3. From the solubility curve for KNO<sub>3</sub>, read the experimental value for the solubility of KNO<sub>3</sub> at 25.0°C. Indicate this reading on your graph by drawing two straight lines: the vertical one corresponds to the temperature of 25.0°C, and the horizontal one indicates the corresponding solubility.
- 4. Calculate the Percentage Error. The theoretical value for the solubility of KNO<sub>3</sub> at 25.0°C is 38.0 g KNO<sub>3</sub> at 25.0°C is 38.0 g KNO<sub>3</sub>/100 g water.

# EXPERIMENT 16: The Effect of Temperature on Solubility REPORT FORM

| NAME  | E:  |                               | Date:                            | Partner(s):  |                                   |
|-------|---|-------------------------------|----------------------------------|--|-----------------------------------|
| Mass  | of vial and KN<br>of vial:<br>of KNO <sub>3</sub> : | O <sub>3</sub> :              | _9                               |  |                                   |
|       |   |                               | DATA TAE                         | BLE  |                                   |
| Trial | Initial Mass<br>of water<br>(g)                     | Mass of<br>Water added<br>(g) | Total<br>Mass of<br>water<br>(g) | SOLUBILITY  (g KNO <sub>3</sub> /100 g H <sub>2</sub> O) | SATURATION<br>TEMPERATURE<br>(°C) |
| 1     | 5.00  | 0.00                          | 5.00                             | 1120)  |                                   |
| 2     | 5.00  | 1.00                          | 6.00                             |  |                                   |
| 3     | 6.00  |                               |                                  |  |                                   |
| 5     |   |                               |                                  |  |                                   |
| 6     |   |                               |                                  |  |                                   |
| 7     |   |                               |                                  |  |                                   |
| 8     |   |                               |                                  |  |                                   |
| 9     |   |                               |                                  |  |                                   |
| 10    |   |                               |                                  |  |                                   |
| 11    |   |                               |                                  |  |                                   |
| 12    |   |                               |                                  |  |                                   |
| 13    |   |                               |                                  |  |                                   |
| 14    |   |                               |                                  |  |                                   |
|       | usion:<br>olubility of KN0                          | O₃ at 25.0 <sup>0</sup> C is  |                                  | _ g KNO₃/100 g wate                                      | r                                 |
|       | <u>nt Error</u><br>imental Value:                   | 9                             | KNO₃/100 g                       | water  |                                   |
| Theor | etical Value:                                       | g                             | g KNO₃/100 g water               |  |                                   |
|       | nt Error:<br>Calculations b                         | pelow:                        | %                                |  |                                   |



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## **EXPERIMENT 17: Chemical Bonding and Molecular Polarity**

#### **PURPOSE:**

- 1. To distinguish between different types of chemical bonds.
- 2. To predict the polarity of some common molecules from a knowledge of bond polarity and molecular geometry.

#### PRINCIPLES:

Isolated atoms are seldom found as such in nature because the majority of atoms are too reactive to exist by themselves. In most substances, atoms are joined together by strong forces called chemical bonds. The way atoms form these chemical bonds is related to their electronic structures and the kind of bonds that exist within compounds is the principal factor determining the chemical properties of these compounds.

In binary **ionic compounds** the two atoms involved in a given ionic bond are a metal and a nonmetal. These atoms are quite different but are complementary to each other; the metallic atom likes to lose electrons and the nonmetallic atom likes to gain electrons. The net result is an electron transfer from the metallic to the nonmetallic atom.

**Covalent bonds**, resulting from electron sharing are formed between similar or identical atoms. In electron sharing, two nuclei attract the same electron and the resulting attractive forces hold the two nuclei together.

At first glance, the ionic and covalent bonds seem to represent distinctively different forms of bonding. Actually the two bonds are the extremes of a broad continuum of bonding patterns. The relationship between the two bonding models is apparent when the concept of electronegativity is considered. **Electronegativity** is a measure or the relative attraction an atom has for the shared electrons in a bond.

The higher the electronegativity value for an element is, the greater the electronattracting ability of the atom for the shared electrons. The difference in the electronegativity values of the atoms in a bond is the key to predicting the polarity of that bond. **Polarity** is a measure or the inequality in the sharing of bonding electrons.

When two identical atoms (atoms of equal electronegativity) share one or more pairs of electrons, each atom exerts the same attraction for the electrons, which results in the electrons being shared equally. This type of bond is called a nonpolar covalent band. A **nonpolar covalent** bond is one in which the sharing of bonding electrons is equal.

When two atoms involved in a covalent bond are not identical (atoms of different electronegativities) the atom that has higher electronegativity attracts the electrons more strongly than the other atom; this results in unequal sharing of electrons. This type of bond is called a polar covalent bond. A **polar covalent** bond is one in which the sharing of bonding electrons is unequal.

It follows that most chemical bonds are neither 100% covalent (equal sharing) nor 100% ionic (no sharing); instead, they f all somewhere in between (unequal sharing).

It is still convenient to use the terms ionic and covalent in describing chemical bonds, based on the following guidelines:

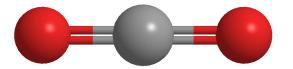
- 1. When there is no difference in electronegativity between bonded atoms, the bond is called a nonpolar covalent bond.
- 2. When the electronegativity difference between bonded atoms is greater than zero but less than 1.7, the bond is called a polar covalent bond.
- 3. When the difference in electronegativity between bonded atoms is 1.7 or greater, the bond is called an ionic bond.

| <b>Electronegativity Difference</b> | Bond Type         |
|-------------------------------------|-------------------|
| Zero                                | Nonpolar Covalent |
| Between 0 and 1.7                   | Polar Covalent    |
| Equal to or greater than 1.7        | lonic             |



When there are three or more atoms bonded together, it is possible to have a nonpolar molecule even though there are polar bonds present. When a molecule contains more than two atoms, we must consider its geometry to decide if the bond is polar. Consider, as a simple example, a molecule AB<sub>2</sub>. Suppose the central atom A is more electronegative than B. Two geometries are possible, bent and linear.





| H₂O  | CO <sub>2</sub>   |
|--|---|
| In this molecule the negative pole is located at the central red oxygen atom. The positive pole is located midway between the white hydrogen atom. | In this molecule the two polar bonds are in exactly opposite directions at 180° angle to each other. The two bond polarities cancel each other out. |
|  |   |
| Polar bond → Polar molecules   | Polar bond → Nonpolar molecules   |
| Non-symmetrical arrangement  | Symmetrical arrangement   |

In predicting the polarity of molecules, the following generalizations might prove useful:

- 1. Molecules containing identical atoms are always non-polar.
- 2. Molecules containing unlike atoms are:
  - i. Nonpolar, if the arrangement of the atoms is symmetrical.
  - ii. Polar, if the arrangement of the atoms is nonsymmetrical.

#### PROCEDURE:

- 1. Assemble the first set of seven models of molecules.
  - a. Use the following colors to represent the atoms:

| Hydrogen | Yellow |
|----------|--------|
| Chlorine | Green  |
| Oxygen   | Red    |
| Nitrogen | Black  |
| Bromine  | Purple |
| Carbon   | Black  |
| Sulfur   | Red    |
| lodine   | Orange |

- b. Use a set of spring connectors for multiple bonds.
- c. Draw the Lewis Electron Dot formula for each formula.
  - i. Use dots to represent both shared and unshared valence electrons.
- d. Evaluate the bond type, note the molecular shape and predict if the molecule is polar or not.
- 2. Assemble the second set of seven molecules. Evaluate the bond types, note the molecular shape and predict if the molecule is polar is not. If the molecule contains more than me type of bond (three different atoms), each bond should be evaluated individually in order to predict if the molecule is polar of not.
- 3. Take the models apart, and place the balls and the connectors in the kit in the same order you have found them at the beginning.

# EXPERIMENT 17: Chemical Bonding and Molecular Polarity REPORT FORM

| NAME: |  | Date: | Partner(s): |  |
|-------|--|-------|-------------|--|
|-------|--|-------|-------------|--|

| Formula         | Electron-Dot Formula | Bond Type    | Shape of Molecule                                       | Kind of<br>Molecule |
|-----------------|----------------------|--------------|---|---------------------|
| H <sub>2</sub>  |                      | 🗖 - Polar    | <ul><li>□ - Linear</li><li>□ - Bent</li></ul>           | □ - Polar           |
| 1 12            |                      | ☐ - Nonpolar | □ - Pyramidal □ - Tetrahedral                           | ☐ - Nonpolar        |
| F <sub>2</sub>  |                      | 🗖 - Polar    | □ - Linear<br>□ - Bent                                  | □ - Polar           |
| 1 2             |                      | 🗖 - Nonpolar | □ - Pyramidal □ - Tetrahedral                           | ☐ - Nonpolar        |
| Br <sub>2</sub> |                      | 🗖 - Polar    | □ - Linear<br>□ - Bent                                  | 🗖 - Polar           |
| Dig             |                      | 🗖 - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | ☐ - Nonpolar        |
| l.              |                      | 🗖 - Polar    | □ - Linear<br>□ - Bent                                  | 🗖 - Polar           |
| l <sub>2</sub>  |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | ☐ - Nonpolar        |
| $N_2$           |                      | 🗖 - Polar    | ☐ - Linear<br>☐ - Bent                                  | 🗖 - Polar           |
| IN <sub>2</sub> |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | 🗖 - Nonpolar        |
| Cl <sub>2</sub> |                      | 🗖 - Polar    | <ul><li>□ - Linear</li><li>□ - Bent</li></ul>           | 🗖 - Polar           |
| OI <sub>2</sub> |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | 🗖 - Nonpolar        |
| HCI             |                      | 🗖 - Polar    | ☐ - Linear<br>☐ - Bent                                  | 🗖 - Polar           |
| 1101            |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | 🗖 - Nonpolar        |
| HBr             |                      | 🗖 - Polar    | ☐ - Linear<br>☐ - Bent                                  | 🗖 - Polar           |
| IIDI            |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | 🗖 - Nonpolar        |
| BrCl            |                      | 🗖 - Polar    | ☐ - Linear<br>☐ - Bent                                  | ☐ - Polar           |
| DICI            |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | 🗖 - Nonpolar        |

| Formula                         | Electron-Dot Formula | Bond Type    | Shape of Molecule                                       | Kind of<br>Molecule |
|---------------------------------|----------------------|--------------|---|---------------------|
|                                 |                      | 🗆 - Polar    | ☐ - Linear<br>☐ - Bent                                  | ☐ - Polar           |
| H <sub>2</sub> O                |                      | □ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | □ - Nonpolar        |
| CO <sub>2</sub>                 |                      | 🗖 - Polar    | ☐ - Linear<br>☐ - Bent                                  | □ - Polar           |
| 002                             |                      | 🗖 - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | □ - Nonpolar        |
| шс                              |                      | 🗆 - Polar    | <ul><li>□ - Linear</li><li>□ - Bent</li></ul>           | □ - Polar           |
| H <sub>2</sub> S                |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | □ - Nonpolar        |
| NH <sub>3</sub>                 |                      | 🗖 - Polar    | <ul><li>□ - Linear</li><li>□ - Bent</li></ul>           | □ - Polar           |
| 14113                           |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | ☐ - Nonpolar        |
| NCI                             |                      | 🗖 - Polar    | <ul><li>□ - Linear</li><li>□ - Bent</li></ul>           | □ - Polar           |
| NCI <sub>3</sub>                |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | ☐ - Nonpolar        |
| CCI                             |                      | 🗖 - Polar    | <ul><li>□ - Linear</li><li>□ - Bent</li></ul>           | □ - Polar           |
| CCI <sub>4</sub>                |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | ☐ - Nonpolar        |
| CH₃CI                           |                      | 🗖 - Polar    | <ul><li>□ - Linear</li><li>□ - Bent</li></ul>           | □ - Polar           |
| CI 13CI                         |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | □ - Nonpolar        |
| CH <sub>2</sub> Cl <sub>2</sub> |                      | 🗖 - Polar    | ☐ - Linear<br>☐ - Bent                                  | □ - Polar           |
| OI 12O12                        |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | ☐ - Nonpolar        |
| CHCl <sub>3</sub>               |                      | 🗖 - Polar    | ☐ - Linear<br>☐ - Bent                                  | 🗖 - Polar           |
| OI 1013                         |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | ☐ - Nonpolar        |
| CH Dr                           |                      | ☐ - Polar    | ☐ - Linear<br>☐ - Bent                                  | ☐ - Polar           |
| CH₃Br                           |                      | ☐ - Nonpolar | <ul><li>□ - Pyramidal</li><li>□ - Tetrahedral</li></ul> | ☐ - Nonpolar        |

### **QUESTIONS**:

- 1. Calculate the electronegativity difference and the percentage ionic character for each of the bonds listed below.
  - a. Use Appendix I for the electronegativity values and percentage ionic character.
  - b. Estimate between the given values of percentage ionic character as needed.

| Bond  | Electronegativity Difference | Percentage of Ionic<br>Character |
|-------|------------------------------|----------------------------------|
| H-O   |                              |                                  |
| H-N   |                              |                                  |
| H-CI  |                              |                                  |
| Br-Cl |                              |                                  |
| H-S   |                              |                                  |
| H-C   |                              |                                  |
| CI-CI |                              |                                  |
| C-O   |                              |                                  |
| K-Br  |                              |                                  |
| Na-O  |                              |                                  |

2. Both water and carbon dioxide are triatomic molecules. Explain why one of these is polar and the other is nonpolar.

3. Classify each of the following molecules as:

| assify each of the fo | pllowing molecules as:                         |
|-----------------------|--|
|                       | ☐ - Ionic Crystal                              |
| Br <sub>2</sub>       | ☐ - Polar Covalent Molecule                    |
|                       | □ - Nonpolar Covalent Molecule                 |
|                       | ☐ - Ionic Crystal                              |
| CCI <sub>4</sub>      | ☐ - Polar Covalent Molecule                    |
| 33.4                  | ☐ - Nonpolar Covalent Molecule                 |
|                       | ☐ - Ionic Crystal                              |
| $CO_2$                | ☐ - Polar Covalent Molecule                    |
| 002                   | ☐ - Nonpolar Covalent Molecule                 |
|                       | ☐ - Ionic Crystal                              |
| BaO                   | ☐ - Polar Covalent Molecule                    |
| Dao                   | ☐ - Nonpolar Covalent Molecule                 |
|                       | □ - Ionic Crystal                              |
| KCI                   | ☐ - Polar Covalent Molecule                    |
| KCI                   |  |
|                       | ☐ - Nonpolar Covalent Molecule                 |
| M=0                   | ☐ - Ionic Crystal                              |
| MgS                   | ☐ - Polar Covalent Molecule                    |
|                       | ☐ - Nonpolar Covalent Molecule                 |
| D 0                   | ☐ - Ionic Crystal                              |
| BeO                   | ☐ - Polar Covalent Molecule                    |
|                       | ☐ - Nonpolar Covalent Molecule                 |
|                       | ☐ - Ionic Crystal                              |
| H <sub>2</sub> O      | ☐ - Polar Covalent Molecule                    |
|                       | ☐ - Nonpolar Covalent Molecule                 |
|                       | ☐ - Ionic Crystal                              |
| AIN                   | ☐ - Polar Covalent Molecule                    |
|                       | ☐ - Nonpolar Covalent Molecule                 |
|                       | ☐ - Ionic Crystal                              |
| LiBr                  | ☐ - Polar Covalent Molecule                    |
|                       | ☐ - Nonpolar Covalent Molecule                 |
|                       | ☐ - Ionic Crystal                              |
| MgO                   | ☐ - Polar Covalent Molecule                    |
|                       | ☐ - Nonpolar Covalent Molecule                 |
|                       | ☐ - Ionic Crystal                              |
| HI                    | □ - Polar Covalent Molecule                    |
|                       | <ul><li>- Nonpolar Covalent Molecule</li></ul> |
|                       | ☐ - Ionic Crystal                              |
| $N_2$                 | □ - Polar Covalent Molecule                    |
|                       | □ - Nonpolar Covalent Molecule                 |
|                       | ☐ - Ionic Crystal                              |
| CaO                   | ☐ - Polar Covalent Molecule                    |
|                       | ☐ - Nonpolar Covalent Molecule                 |
|                       | ☐ - Ionic Crystal                              |
| CO                    | ☐ - Polar Covalent Molecule                    |
|                       | ☐ - Nonpolar Covalent Molecule                 |
|                       |  |



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# **EXPERIMENT 18: Crystal Structure**

#### PURPOSE:

- 1. To analyze models of various unit cells for crystalline solids.
- 2. To calculate the fraction of the unit cell volume that is empty space in a crystalline solid.
- 3. To study models of the unit cells of various ionic compounds and show the relationship of the number of ions in the unit cell to the simplest formula.
- 4. To determine the number of atoms within the unit cell of a covalent network solid.

#### Principles:

Crystalline solids are characterized by distinctive geometric shapes, which arise from the definite orderly patterns of the constituent atoms, ions or molecules. In studying the architecture of these crystalline materials, it is helpful to introduce a geometrical idealization. We substitute points fro the atoms, ions or molecules, placing points where their centers would be. This regular array of points in space is called a **space lattice**. A regular network can connect the lattice points, thus dividing the lattice into a number of **unit cells**.

The unit cell is defined as the smallest portion of the space lattice which, when moved a distance equal to its own dimensions in various directions, generates the whole space lattice. The simplest space lattices are based upon cubic unit cells and will be studied in some detail in this experiment.

#### Procedure:

#### I. SIMPLE CUBIC LATTICE

- 1. Locate the preassembled model containing three sets of nine spheres of equal size in a square arrangement. This model contains a colored center sphere.
  - i. Remove the top layer of nine spheres by sliding the spheres upward the metal rods.



DO NOT REMOVE THE METAL RODS FROM THE PLASTIC BASE. PLACE THE NINE SPHERES YOU HAVE REMOVED IN A SMALL BEAKER. DO NOT LOSE ANY OF THE SPHERES.



- ii. Study this model and determine the number of unit cells formed by the two sets of nine spheres. Record your answer in your Laboratory Notebook.
- iii. Note that the center colored sphere of the upper layer is in common with all of the unit cells.
- iv. Place the third set of nine spheres you have previously set aside on the top of the second set. Study the total arrangement. Enter the total number of unit cells formed by the three sets of spheres in your Laboratory Notebook. Note that the colored sphere at the center is in common with all of the simple cubic unit cells.
- v. What fraction of this colored sphere (that lies at a corner of all these simple cubic unit cells) lies within any one of the unit cells? Enter your answer in your Laboratory Notebook. Observe that any sphere lying at the

- corner of a cubic unit cell contributes this same fraction of a sphere to the unit cell.
- vi. The equivalent of how many spheres are within a simple cubic unit cell? Record this value in your Laboratory Notebook.
- 2. Locate the preassembled model of a simple cubic model containing 12 clear spheres.
  - i. Note that there are 3 layers of four clear plastic spheres each, in a square arrangement.
  - ii. Remove the top layer of four spheres by sliding the spheres upward the metal rods.



DO NOT REMOVE THE METAL RODS FROM THE PLASTIC BASE.
PLACE THE NINE SPHERES YOU HAVE REMOVED IN A SMALL BEAKER.
DO NOT LOSE ANY OF THE SPHERES.



iii. Your model now consists of two sets of four spheres attached in a square arrangement.

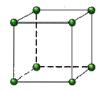


Figure 1

- iv. The dots in Figure 1 represent the centers of the atoms.
- v. In your model the spheres should be in contact. This represents one unit cell.
- vi. By using the four spheres that you have previously set aside, extend your model upward so that you form two unit cells in contact.

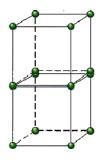
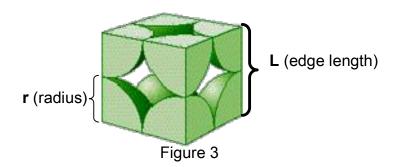


Figure 2

- vii. Note that two unit cells share four of your spheres (middle layer).
- viii. Focus you attention on one unit cell. Note that, because the spheres are in contact along the cube edge, the edge length of the unit cell (distance from sphere center to sphere center) is just twice the radius of one sphere. See Figure 3.

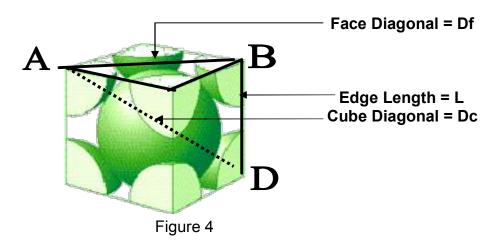


- ix. Record the edge length, **L**, in terms of the sphere radius, **r**, in your Laboratory Notebook.
- x. Determine the volume of the unit cell (in terms of  $\mathbf{r}^3$ ).
- xi. Determine the total volume of all spheres within the unit cell in terms of  $\bf r$  and express your answer as a decimal:  $\bf a \times r^3$ . It is convenient to assign to "a" three significant figures. (Volume of a sphere =  $\bf 4 \times r^3/3$  Use  $\pi = \bf 3.14$ )
- xii. Calculate the fraction of the unit cell that is empty space and express your answer as a percentage in two significant figures.

% Empty Space = 
$$\frac{(Volume\ of\ the\ unit\ cell) - (Volume\ occupied\ by\ the\ spheres)}{(Volume\ of\ the\ unit\ cell)} \times 100\%$$

#### II. BODY-CENTERED-CUBIC LATTICE

- Locate the preassembled model containing a single unit cell of the bodycentered-cubic lattice.
  - I. Record the equivalent number of spheres within the unit cell.



- II. Calculate the edge length, L, of the unit cell in terms of sphere radius, r. Hints:
  - a. Record the cube Diagonal length (Dc) in terms of sphere radius (r)
  - b. Note that the spheres are in contact along the cube diagonal but not along the cube edges.

B. Using triangle ABC, express  $Df^2$  as a function of  $L^2$  (Equation 1) Note that : AC = BC = L and AB = Df

- C. Using triangle ABD, express  $Dc^2$  as a function of  $Df^2$  and  $L^2$ . (Equation 2) Note that AD = Dc AB = Df and BD = L
- D. In Equation 2 express **Dc** in terms of **r** and replace the value of **Df** with that obtained in Equation 1.

Solve for L, in terms of r.

## 3. Determine the volume of the unit cell (in terms of $r^3$ ).

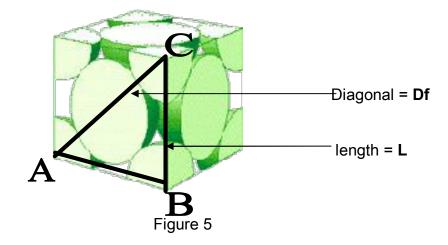
Express your answer as a decimal: **b** x **r**<sup>3</sup>. It is convenient to assign to "**b**" three significant figures.

- 4. <u>Determine the total volume of all spheres within the unit cell (in terms of  $r^3$ )</u> Express your answer as a decimal:  $\mathbf{c} \times \mathbf{r}^3$ . It is convenient to assign to " $\mathbf{c}$ " three significant figures. (Volume of a sphere =  $4 \pi r^3/3$  Use  $\pi = 3.14$ )
- **5.** Calculate the fraction of the unit cell that is empty space. (See Part I.2) Express your answer as a percentage in two significant figures.

#### III. FACE-CENTERED-CUBIC LATTICE

<u>Locate the preassembled model containing a single unit cell of the body-centered-cubic lattice.</u>

1. Record the equivalent number of spheres within the unit cell.



### 2. Calculate the edge length, I, of the unit cell in terms of sphere radius, r.

Hints: A. Express Df as a a function of r.

Note that the spheres are in contact along the face diagonal.

- B. Using triangle  $\dot{A}BC$ , express  $Df^2$  as a function of  $L^2$  (Equation 3)
- C. In Equation 3 express **Df** in terms of **r**. Solve for **L**, in terms of **r**.

## 3. Determine the volume of the unit cell (in terms of $r^3$ ).

Express your answer as a decimal: **d x r**<sup>3</sup>. It is convenient to assign to "**d**" three significant figures.

- 4. <u>Determine the total volume of all spheres within the unit cell (in terms of  $r^3$ )</u> Express your answer as a decimal:  $e \times r^3$ . It is convenient to assign to "e" three significant figures. (Volume of a sphere =  $4 \pi r^3/3$  Use  $\pi = 3.14$ )
- 5. Calculate the fraction of the unit cell that is empty space.

Express your answer as a percentage in two significant figures.

#### IV. SODIUM CHLORIDE STRUCTURE

Locate the preassembled model containing a single unit cell of sodium chloride

The smaller spheres (blue) represent the Na<sup>+</sup> caions and the larger colorless spheres represent the Cl<sup>-</sup> anions.

- 1. Record the equivalent number of Na<sup>+</sup> cations within the unit cell
- 2. Record the equivalent number of Cl<sup>-</sup> anions within the unit cell
- 3. Record the simplest ratio of Na<sup>+</sup> cations to Cl<sup>-</sup> anions in the unit cell
- 4. Write the simplest formula of sodium chloride

#### V. CUPRITE STRUCTURE

Locate the preassembled model containing a single unit cell of cuprite

Cuprite is a compound of copper cations (blue spheres) and oxide anions (colorless spheres)

Study the model and note that the oxide anions have a body-centered-cubic arrangement.

- 1. Record the equivalent number of Copper cations within the unit cell
- 2. Record the equivalent number of Oxide anions within the unit cell
- 3. Record the simplest ratio of Copper cations to Oxide anions in the unit cell
- 4. Write the simplest formula of Cuprite

#### VI. SPHALERITE STRUCTURE

#### Locate the preassembled model containing a single unit cell of sphalerite

Sphalerite is a compound of zinc cations (blue spheres) and sulfide anions (colorless Or yellow spheres).

Study the model and note that the sulfide anions form a face-centered-cubic arrangement.

- 1. Record the equivalent number of Zinc cations within the unit cell
- 2. Record the equivalent number of Sulfide anions within the unit cell
- 3. Record the simplest ratio of Zinc cations to Sulfide anions in the unit cell
- 4. Write the simplest formula of Sphalerite

#### VII. DIAMOND STRUCTURE

#### Locate the preassembled model containing a single unit cell of diamond

Diamond is a covalent network solid made of carbon atoms represented by black spheres.

Study the unit cell and determine the number of carbon atoms within the unit cell.

|   |                  | Name:    |
|---|------------------|----------|
|   | Partner:         | _        |
| REPORT FORM   |                  |          |
| . <u>SIMPLE CUBIC LATTICE</u> Number of unit cells formed by two sets of nine | spheres:         | <u> </u> |
| Number of unit cells formed by three sets of nin                              | e spheres:       |          |
| Fraction of a corner sphere that lies within the c                            | cubic unit cell: |          |
| Total number of spheres within one simple cubi                                | c cell:          |          |
| Edge length, I, of the unit cell in terms of r:                               |                  | _        |
| Volume of the unit cell in terms of $\mathbf{r}^3$ :                          |                  | _        |
| Volume of the spheres within the unit cell:                                   |                  | _        |
| Fraction of empty space within the unit cell (%): Show calculation below:     | :                |          |
|   |                  |          |

| Ch | emistry | 1 | O. | 1 |
|----|---------|---|----|---|
|    |         |   |    |   |

ΙΙ.

| BODY-CENTERED-CUBIC LATTICE Total number of spheres within the unit cell:                     |  |
|---|--|
| Diagonal length (Cube diagonal = <b>Dc</b> ) in units of <b>r</b> :                           |  |
| <b>Df</b> <sup>2</sup> (Face diagonal squared) in terms of <b>I</b> <sup>2</sup> :            |  |
| Dc² (Cube diagonal) in terms of Df² and I²  |  |
| Edge length, <b>I</b> , in terms of <b>r</b> : Show calculations below:                       |  |
|   |  |
|   |  |
| Volume of the unit cell in terms of <b>r</b> <sup>3</sup> : Show calculations below:          |  |
|   |  |
| Volume of the spheres within the unit cell in terms of ${\bf r}^3$ : Show calculations below: |  |

|     | Fraction of empty space within the unit cell (%): Show calculations below:                    |  |
|-----|---|--|
|     |   |  |
|     |   |  |
| II. | FACE-CENTERED-CUBIC LATTICE Total number of spheres within the unit cell:                     |  |
|     | Edge length, <b>I</b> , of the unit cell in terms of <b>r</b> : Show calculations below:      |  |
|     |   |  |
|     | Volume of the unit cell in terms of <b>r</b> <sup>3</sup> Show calculations below:            |  |
|     | Volume of the spheres within the unit cell in terms of ${\bf r}^3$ : Show calculations below: |  |
|     | Fraction of empty space within the unit cell (%);   |  |
|     | Show calculations below:  |  |

| Chemistry | 1 | 01 | 1 |
|-----------|---|----|---|
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| IV.         | SODIUM CHLORIDE STRUCTURE  Number of sodium cations within the unit cell:   |
|-------------|---|
|             | Number of chloride anions within the unit cell:   |
|             | Simplest ratio of sodium cations to chloride anions in unit cell:   |
|             | Simplest formula of sodium chloride:  |
| V. <u>.</u> | CUPRITE STRUCTURE  Number of copper cations within the unit cell:   |
|             | Number of oxide anions within the unit cell:  |
|             | Simplest ratio of copper cations to oxide anions in unit cell:  |
|             | Simplest formula of cuprite:  |
| VI.         | SPHALERITE STRUCTURE  Number of zinc cations within the unit cell:  |
|             | Number of sulfide anions within the unit cell:  |
|             | Simplest ratio of zinc cations to sulfide anions in unit cell:  |
|             | Simplest formula of sphalerite:   |
| VII         | . <u>DIAMOND STRUCTURE</u> Number of carbon atoms within the unit cell:   |
| If th       | ercise:<br>ne argon atom has a radius of 1.54 A, what fraction of an argon gas sample at STP actually empty space? <b>Please express all volumes in cm</b> <sup>3</sup> . |



## **EXPERIMENT 30: Percentage of Copper in Malachite**

#### **PURPOSE**:

- 1. To determine the percentage of copper in the naturally occurring copper ore (malachite).
- 2. To determine the relative number of moles of copper atoms in malachite.

#### **PRINCIPLES:**

Malachite is a naturally occurring green mineral used as an ore of copper or for ornamental objects. The chemical name of malachite is basic copper (II) carbonate, and its formula is CuCO<sub>3</sub>•Cu(OH)<sub>2</sub>.

The most familiar form of malachite is the green coating that forms on copper objects, such as statues and buildings as the result of prolonged weathering. This green film acts or patina acts as a protective coating and as a result the copper object covered with a layer of malachite that resists further corrosion, as long as this layer is not removed.

The deliberate production of coatings such as malachite is called artificial antiquing and is a technique used in interior decorating. Natural antiquing is often prevented by coating the copper surface with a thin layer of plastic. As long as the layer of plastic is not removed by a zealous housekeeper with vigorous cleaning, the copper will be protected and no malachite will form on its surface.

#### METHOD:

In this experiment the percentage of copper in basic copper (II) carbonate, CuCO<sub>3</sub>•Cu(OH)<sub>2</sub> will be determined experimentally by the thermal decomposition of the compound. The experimental result will then be compared with the theoretical value calculated from the formula.

From the experimentally obtained percentage of copper in malachite, the number of moles of copper per one mole of malachite will be calculated. When basic copper (II) carbonate (malachite) is heated to high temperatures (above 400°C), it decomposes according to the following equation:

$$CuCO_3 \cdot Cu(OH)_{2(s)} \rightarrow 2CuO_{(s)} + CO_{2(g)} + H_2O_{(g)}$$
  
green malachite black solid residue

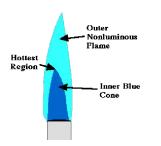
The method used here is to mass a quantity of basic copper (II) carbonate, heat it to drive off the carbon dioxide and the water vapor and then mass the residue, which is assumed to be copper (II) oxide.

In a thermal decomposition such as this, the product must be "heated to constant mass" before you can be sure the decomposition of the malachite is complete. After the first heating, cooling and massing of the decomposed product, the crucible is heated a second time and is cooled and massed again.

If the two masses are the same (within the experimental limits of the equipment used or 0.02 g in this case) it may be assumed that all of the carbon dioxide and all of the water vapor was removed in the first heating. If the two masses are not the same, it must be assumed that the first decomposition was incomplete and some of the original basic copper (II) carbonate may still be present after the second heating. The procedure is repeated as **many times as necessary** until there is no further mass loss. This is where the expression "heating to constant mass" comes from.

#### PROCEDURE:

1. Place a clean, dry crucible on a clay triangle and heat for two or three minutes at maximum flame temperature. The tip



of the sharply defined inner blue cone of the flame (hottest part) should almost touch the crucible bottom.



2. Allow the crucible to cool to room temperature.

NOTE:

Handle the crucible with tongs. Grasp crucible with the concave part of the tongs below the rim of the crucible and handle carefully. It is advisable to hold a watch glass or wire gauze under the crucible while transferring the sample to the balance as a precaution.

- 3. Mass the cooled crucible to the nearest 0.0 1 g.
- 4. Add between 2.3 and 2.5 g of basic copper (II) carbonate.
- 5. Mass again to the nearest 0.01 g.
- 6. Place the crucible on the clay triangle. One more time, adjust the flame so, that the tip of the sharply defined inner blue cone almost touches the bottom of the crucible.
- 7. Heat strongly for five minutes, the bottom of the crucible should be heated to a dull red color during this period.



- 8. Allow the crucible to cool to room temperature (a minimum of ten minutes).
- 9. Mass the cool crucible containing the residue to the nearest 0.01 g.
- 10. Heat the crucible again, strongly for another five minutes at maximum flame temperature (bottom of the crucible heated to a dull red color)
- 11. Cool the crucible and contents to room temperature (a minimum of ten minutes).
- 12. Remass to the nearest 0.01 g.

NOTE: IF THE TWO MASSINGS DO NOT AGREE TO ABOUT 0.02 g, REPEAT THE HEATING UNTIL CONSTANT MASS IS ACHEIVED.



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# Experiment 30: PERCENTAGE OF COPPER IN MALACHITE REPORT FORM

| of Copper in CuCO <u>3</u> •Cı          |                          |  |
|---|--------------------------|--|
|   | <u>ı(OH)<sub>2</sub></u> |  |
| ıCO <sub>3</sub> •Cu(OH) <sub>2</sub> : | g                        |  |
| le:                                     | g                        |  |
| )H) <sub>2</sub> :                      | g                        |  |
| ıO (first heating)                      | :                        | g  |
|   |                          |  |
|   |                          |  |
|   |                          |  |
|   | g                        |  |
|   | 9                        |  |
|   | rimental):               | %  |
| CuCO3•Cu(OH)2 (theor<br>below:          | etical):                 | %  |
|   | ole:                     | (second heating):  (third heating):  (DO (best):  (Second heating):  (third heating):  (DO (best):  (DO (best |

| PART II: Relative Number of Copper Atoms in Malachite                             |   |
|---|---|
| Mass of CuCO <sub>3</sub> •Cu(OH) <sub>2</sub> :                                  | g |
| Mass of 1 mole of CuCO <sub>3</sub> •Cu(OH) <sub>2</sub> :                        |   |
| Number of moles of CuCO <sub>3</sub> •Cu(OH) <sub>2</sub> :                       |   |
| Show calculations below:  |   |
| Mass of Cu (experimental, from PART I):   |   |
| Mass of 1 mole of Cu:   | g |
| Number of moles of Cu:  |   |
| Show calculations below:  |   |
| Experimental mole ratio: $\frac{moles\ of\ Cu}{moles\ of\ CuCO_3 \cdot Cu(OH)_2}$ |   |
| Theoretical mole ratio: $\frac{moles\ of\ Cu}{moles\ of\ CuCO_3 \cdot Cu(OH)_2}$  |   |
| moles of $CuCO_3 \cdot Cu(OH)_2$ Percentage error                                 |   |

Percentage error

Show calculations below:

### **EXPERIMENT 31: Table Salt from Soda Ash**

#### **PURPOSE:**

- 1. To obtain sodium chloride from sodium hydrogen carbonate
- 2. To study the stoichiometry of this reaction.

#### METHOD:

Baking soda is the common name for sodium hydrogen carbonate, NaHCO<sub>3</sub>. When NaHCO<sub>3</sub> is treated with hydrochloric acid, HCl<sub>(aq)</sub>, it produces a white solid residue, sodium chloride. Sodium chloride is commonly called table salt. The reaction also produces two gaseous products, water vapor and carbon dioxide.

$$NaHCO_{3(s)} + HCl_{(aq)} \rightarrow NaCl_{(s)} + H_2O_{(g)} + CO_{2(g)}$$

#### PROCEDURE:

- 1. Mass a clean, dry, empty crucible on the centigram balance.
- 2. Remove the crucible from the balance and add approximately 1.0g-1.5g of NaHCO<sub>3</sub> into the crucible.



### DO NOT EXCEED THE AMOUNT OF NaHCO<sub>3</sub> INDICATED.



- 3. Determine the exact mass of the crucible and its content on the centigram balance.
- 4. Determine the exact mass of the NaHCO<sub>3</sub> in the crucible by difference.
- 5. In the fume hood, measure out 4-5 mL of concentrated hydrochloric acid (12 M) in your small graduated cylinder. Record this volume to the nearest 0.1 mL (You must measure out at least 4.0 mL)



# CAUTION! CONCENTRATED HYDROCHLORIC ACID IS HIGHLY CORROSIVE AND GIVES OFF NOXIOUS FUMES!



Transfer the concentrated hydrochloric acid to a small beaker and cover it with a watch glass. Leave the beaker under the fume hood



DO NOT RETURN ANY CONCENTRATED HYDROCHLORIC ACID TO THE ORIGINAL REAGENT BOTTLE.





DO NOT REMOVE THE CONTAINER WITH HYDROCHLORIC ACID FROM THE FUME HOOD.



If you measured out too much concentrated hydrochloric acid, dispose of it into an appropriately labeled waste container found in the fume hood.

7. In the fume hood, add drop wise (use a Pasteur pipette) the concentrated hydrochloric acid to the sample and observe the effervescence.

#### NOTES:

- If the effervescence is too vigorous, slow down the rate at which the concentrated hydrochloric acid is added.
- To avoid splattering of the sample, slow down the rate at which the concentrated hydrochloric acid is added.
- This step is completed when all of the concentrated hydrochloric acid has been added to the sample.
- 8. Heat the crucible under the fume hood until the sample appears dry.
- 9. Remove the crucible from the hood and continue heating at your bench until constant mass is achieved when successive massings agree within 0.01 g.



If you detect any trace of objectionable fumes given off by the sample, return the crucible immediately to the fume hood to resume heating.





If the residue starts to melt (glassy appearance), this indicates that the heating is too strong and the residue had probably been already heated to dryness and hence constant mass.





Keep In mind that heating is done for the sole purpose to completely drive off the gaseous products, and not to melt the residue.



10. Record the mass of the crucible and the residue (constant mass) and determine the mass of the residue.

# Experiment 31: Table Salt from Soda Ash REPORT FORM

| NAME:                             | Date:                                    | Partner(s):                    |          |
|-----------------------------------|--|--------------------------------|----------|
| <u>Data:</u><br>Mass of crucible: |  | g                              |          |
| Mass of crucible and              | sample Na <sub>2</sub> CO <sub>3</sub> : | g                              |          |
| Mass of sample Na <sub>2</sub> C  | O <sub>3</sub> :                         | g                              |          |
| Massif crucible + resi            | due:                                     | g                              |          |
| Mass of residue:                  |  | g                              |          |
| Volume of conc. HCI               | added                                    | mL                             |          |
| <u>Calculations</u>               |  |                                |          |
| Mass of 1 mole of Na              | <sub>2</sub> CO <sub>3</sub>             | g/mole                         |          |
| Number of moles of N              | la₂CO₃ added                             | moles                          |          |
| Concentration of HCI              | aq)                                      | <u>12</u> M                    |          |
| Number of moles of H              | ICI added                                | moles                          |          |
| Write a balanced che              | mical equation for this ch               | emical reaction. Include state | •        |
| designations for all re           | actants and products.                    |                                |          |
|                                   |  |                                |          |
| How many moles of H               | ICI are required to react (              | completely the quantity of Na  | CO2 VOU  |
| •                                 | ior are required to react                |                                | ,003 you |
| naa measarea out: _               |  | 10103 1101                     |          |
| Which reactant is limi            | ting?                                    |                                |          |
| Which reactant is in e            | xcess?                                   |                                |          |

| Chemistry 101             |   |  |
|---------------------------|---|--|
| Actual yield of NaCl      | g |  |
| Theoretical yield of NaCl | g |  |
| Show calculations below:  |   |  |
|                           |   |  |
|                           |   |  |
|                           |   |  |
|                           |   |  |
|                           |   |  |
| Percent yield of NaCl     | % |  |
| Show calculations below:  |   |  |

# EXPERIMENT 32: Equivalent Mass Determination in Oxidation-Reduction Reactions

#### **PURPOSE:**

 To determine the Equivalent Mass of an Unknown Reducing Agent by titration with a standard solution of 0.1 N potassium permanganate

#### **PRINCIPLES**:

Chemical reactions in which the oxidation states of two or more elements in the ions or molecules of the reactants are changed are called oxidation-reduction reactions.

Changes in oxidation states are brought about by a transfer of electrons; an ion or molecule which gains electrons is reduced (less positive oxidation state) and is called an oxidizing agent since the co-reactant which provided the reducing electrons has been oxidized (lost electrons - higher oxidation state). Similarly, the co-reactant is called a reducing agent since it is oxidized in the reaction.

In order to simplify stoichiometric calculations in oxidation-reduction reactions, we define reactant and product masses in terms of the amount of substance which gains or loses one mole of electrons in a given reaction.

The amount of substance which gains or loses one mole of electron in a given oxidation-reduction reaction is called the equivalent mass of the substance.

For consistency, the solution concentrations are expressed in units of equivalents/liter of solution or Normality (N).

The oxidation-reduction reaction shown below involves the reducing agent,  $H_2C_2O_4$ , oxalic acid.

#### **Overall Reaction:**

$$3H_2SO_4 + 2KMnO_4 + 5H_2C_2O_4 \rightarrow 10CO_2 + 2MnSO_4 + K_2SO_4 + 8H_2O_4$$

#### Half Reactions:

$$MnO_{-4}^{-} - 8H^{+} + 5e^{-} \rightarrow Mn^{2+} + 4H_{2}O$$
  
 $H_{2}C_{2}O_{4} \rightarrow 2CO_{2} + 2H^{+} + 2e^{-}$ 

#### Half Reactions (to balance):

$$2 \times \left(MnO_{4}^{-} - 8H^{+} + 5e^{-} \to Mn^{2+} + 4H_{2}O\right)$$
$$5 \times \left(H_{2}C_{2}O_{4} \to 2CO_{2} + 2H^{+} + 2e^{-}\right)$$

$$2MnO_4^- + 5H_2C_2O_4^- + 6H^+ \rightarrow 10CO_2^- + 2Mn^{2+} + 8H_2O_2^-$$

The solid acid (oxalic acid,  $H_2C_2O_4$ ) is used as a primary standard since it can be made with good accuracy. This is not the case for KMnO<sub>4</sub> which has impurities that are not easily removed and is not very stable toward reactions with substances in the environment.

The manganese in the permanganate ion  $(Mn^{7+})$  gains five electrons in converting to  $MnSO_4$   $(Mn^{2+})$ . Since each mole of  $KMnO_4$  (157 g/mole) dissolved in solution gives one mole of  $MnO_4$  which **gains five moles of electrons** in the reaction, its equivalent mass (31.4 g/equivalent) is **one-fifth** its molar mass.

Similarly oxalic acid (90.0 g/mole) which **loses two moles of electrons** per mole of acid has an equivalent mass of **one-half** of its molar mass (45.0 g/equivalent).

The equivalent mass of an oxidizing or reducing agent always refers to a specific oxidation reduction reaction.

#### PROCEDURE:

- 1. Obtain 100 mL of standard 0.1 N KMnO₄ in a clean, dry 250 mL Erlenmeyer flask. Keep the flask stoppered.
- 2. Record the exact normality of the KMnO<sub>4</sub> solution
- 3. Rinse a clean buret with three 5 mL portions of KMnO<sub>4</sub> solution.
- Fill the buret near the zero mark. Since the solution is opaque, you must read
  the volume level at the top of the solution rather than at the bottom of the
  meniscus.
- 5. The instructor will provide a vial containing an unknown reducing agent whose equivalent mass will be determined.
- 6. Record your unknown number.
- 7. Mass the vial including the sample and then pour out about 0.2 g of your unknown directly into a 250 mL Erlenmeyer flask.
- 8. Record the mass of the vial without the sample. (The mass of the sample is obtained by difference.)
- 9. Repeat two more times to give three samples (a total of four massings is required.)
- 10. Add 30 mL of 3M H<sub>2</sub>SO<sub>4</sub> and 75 mL deionized water to each flask.

- 11. To titrate your first sample, heat the solution in the flask to just below boiling and <u>titrate while hot</u> to a pale pink color which persists after stirring. (The KMnO<sub>4</sub> solution serves as its own indicator in the titration.)
- 12. Repeat the titration process with the other two samples you have prepared.

#### NOTES:

- At the start of the titration, the permanganate color will disappear slowly (several seconds) as the KMnO<sub>4</sub> solution is added.
- As the titration proceeds, Mn<sup>2+</sup> Is produced which catalyzes the reaction.
- The end point occurs when the pink color persists after the addition of one drop of KMnO<sub>4</sub> solution.
- The solution should be hot at the end point, otherwise the reaction will be too slow and high results will be obtained.
- 13. Calculate the equivalent mass for each sample titrated and the value of the average equivalent mass.



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# Experiment 32: Equivalent Mass Determination in Oxidation-Reduction Reactions REPORT FORM

|   |                              | KLFOKIII | <u>OKIVI</u> |       |       |
|---|------------------------------|----------|--------------|-------|-------|
| Ν | AME:                         | Date:    | Partner(     | s):   |       |
| Ν | ormality of KMnO₄ solution:  |          | N            |       |       |
| D | ATA:                         |          |              |       |       |
|   |                              | First    | Second       | Third | Forth |
|   | Initial mass, vial + unknown | g        | g            | g     |       |
|   | Final mass, vial + unknown   |          |              |       |       |

## **CALCULATIONS:**

Mass of unknown

Final buret reading (KMnO<sub>4</sub>)

Initial buret reading (KMnO<sub>4</sub>)

Volume of KMnO<sub>4</sub> used

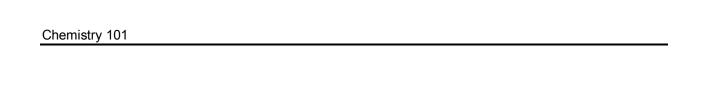
Equivalents of KMn04 = Equivalents of Unknown = Liters KMnO<sub>4</sub> x Normality

|  | First | Second | Third | Forth |
|--|-------|--------|-------|-------|
| Equivalents of KMnO <sub>4</sub> (and of unknown) in the sample titrated | eq    | eq     | eq    | eq    |

mL

 $Equivalent \ mass \ of \ unknown = \frac{grams \ of \ unknown}{equivalents \ of \ unknown}$ 

|  | First | Second | Third | Forth |
|--|-------|--------|-------|-------|
| Mass of one equivalent of unknown (g/equivalent) |       |        |       |       |
| Average Value (g/equivalent)                     |       |        |       |       |
| Deviation  d <sub>i</sub>   (g/equivalent)       |       |        |       |       |
| Mead Deviation,                                  |       |        |       |       |
| $\overline{d}$ (g/equivalent)                    |       |        |       |       |
| Relative Mean Deviation,                         |       |        |       |       |
| RMD  |       |        |       |       |



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# **EXPERIMENT 33: Standardization of a Sodium Hydroxide**Solution with a Primary Standard

#### PURPOSE:

• To standardize a solution of sodium hydroxide by titration with a primary standard (KHC<sub>8</sub>H<sub>4</sub>O<sub>4</sub>), potassium acid phthalate.

#### **PRINCIPLES**:

Acid-base reactions in aqueous solution represent one of the most important types of chemical reactions. For many years **acids** were described as:

- Substances that tastes sour.
- Substances that turn blue litmus paper red.
- o Substances that react with metals to produce hydrogen gas.
- Substances that react with carbonates and bicarbonates to produce carbon dioxide.
- Substances that react with bases to form water and a salt.

#### Similarly **bases** were described as:

- Substances that tastes bitter.
- Substances that feels soapy or slippery to touch.
- o Substances that turn red litmus paper blue.
- o Substances that react with acids to produce water and a salt.

In 1887, Swedish chemist Svante Arrhenius showed that acids are compounds containing hydrogen atoms that can ionize in water to form hydrogen ions. These substances are now known as **Arrhenius acids**. A solution containing excess hydrogen ions is an acid solution, and the more hydrogen ions present, the more acidic the solution.

More recent studies indicate that the hydrogen ion or proton in aqueous solution is hydrated, producing a new species called the hydronium ion  $(H_3O^+)$ . Pure water already contains small quantities of the  $H^+$  ion (or  $H_3O^+$ ) due to the **autoionization** reaction, written either as:

$$\begin{split} H_2O_{(l)} &\longleftrightarrow H^+ + OH^- \\ &\text{or} \\ H_2O_{(l)} + H_2O_{(l)} &\longleftrightarrow H_3O^+ + OH^- \end{split}$$

Acid substances may also be defined as proton (H<sup>+</sup>) donors within the context of a given reaction, according to the description of Brönsted and Lowry (1932):

$$HCl_{(aq)} + H_2O_{(l)} \to H_3O^+ + Cl^-$$

These are known as **Brönsted-Lowry** acids. Whenever solutions of aqueous acids are discussed, the acid species in the solutions is the hydronium ion.

**Arrhenius bases** are defined most simply as substances that generate hydroxide ions in solution:

$$NaOH_{(aa)} \xrightarrow{H_2O} K^+ + OH^-$$

Brönsted and Lowry expanded the definition of a base to include any species that can accept a proton; such substances are row called Brönsted-Lowry bases. Thus, all hydroxides are bases, as are carbonates, bicarbonates, acetates, and ammonia.

$$OH^{-} + H^{+} \rightarrow H_{2}O$$

$$CO_{3}^{2-} + H^{+} \rightarrow HCO_{3}^{-}$$

$$HCO_{3}^{-} + H^{+} \rightarrow H_{2}CO_{3}$$

$$C_{2}H_{3}O_{2}^{-} + H^{+} \rightarrow HC_{2}H_{3}O_{2}$$

$$NH_{3} + H^{+} \rightarrow NH_{4}^{+}$$

The reaction between an Arrhenius acid and an Arrhenius base or between a Brönsted-Lowry acid-base pair is called a **neutralization reaction**. In a complete neutralization in aqueous solution, the  $H_3O^+$  ions react with an equal number of  $OH^-$  ions. The neutralization of an acid releasing one  $H_3O^+$  per formula unit (**monoprotic acid**) with a base releasing one  $OH^-$  (**monohydroxy** or **monobasic base**) exhibits a simple stoichiometry.

An example of such a reaction is illustrated by the equation describing the reaction of hydrochloric acid with sodium hydroxide:

$$HCl_{(aq)} + NaOH_{(aq)} \rightarrow NaCl_{(aq)} + H_2O_{(l)}$$

The complete neutralization requires the moles of acid  $(n_a)$  to be equal to the moles of base  $(n_b)$ :

$$\frac{n_a}{n_b} = \frac{1}{1}$$

or

$$n_a = n_b$$

These relationships can be combined with the definition of molarity:

$$\begin{split} n_a &= M_a \times V_a \\ n_b &= M_b \times V_b \\ M_a \times V_a &= M_b \times V_b \end{split}$$

The last relationship applies whenever the acid and base react in a 1:1 mole ratio. The reaction of diprotic sulfuric acid with dibasic calcium hydroxide is described similarly:

$$H_2SO_{4(aq)} + Ca(OH)_{2(aq)} \rightarrow CaSO_{4(aq)} + H_2O_{(l)}$$
 
$$\frac{n_a}{n_b} = \frac{2}{2} = \frac{1}{1}$$

$$M_a \times V_a = M_b \times V_b$$

The stoichiometry becomes more complex when the acid and base release unequal numbers of hydronium and hydroxide ions. The neutralization of sulfuric acid with sodium hydroxide is expressed by the balanced equation:

$$H_2SO_{4(aq)} + 2NaOH_{(aq)} \rightarrow Na_2SO_{4(aq)} + 2H_2O_{(l)}$$

The 1:2 mole ratio of the acid to base in the balanced equation indicates that twice as many moles of base are needed than of acid:

$$\frac{n_a}{n_b} = \frac{1}{2}$$

$$2n_a=n_b$$

$$2(M_a \times V_a) = M_b \times V_b$$

The quantitative relationships between the amounts of acid and base consumed in a neutralization reaction are treated as simple stoichiometry problems, or better yet in terms of **equivalents**.

By definition, **one gram-equivalent of an acid** is that mass of the acid in grams that will provide 1 mol of protons (H<sup>+</sup>) in a reaction.

A gram equivalent of a base is defined as that mass of the base in grams that will provide 1 mol of hydroxide ions (OH<sup>-</sup>) in a reaction, or that will react with I mol of protons.

A gram equivalent weight is often referred to as an equivalent (equiv.). In acid-base reactions, 1 equivalent of an acid will react with 1 equivalent of a base.

Thus 1 mol of HCI (36.5 g) is 1 equivalent weight of HCI. However, 1 mol of  $H_2SO_4$  reacts with 2 mol of NaOH, and 2 moles of protons are transferred from the acid to the base. Therefore, 1 mol of  $H_2SO_4$ , (98.0 g) corresponds to 2 gram-equivalent weights. In other words, 1 gram-equivalent of  $H_2SO_4$ , or 49.0 g, is equal to 1/2 mol of the acid (2 equiv  $H_2SO_4$ /1 mol  $H_2SO_4$ ).

For volumetric work, **normality** is the most convenient method of expressing concentrations.

Normality, N, is defined as the number of equivalents of solute in a liter of solution.

A useful mathematical relationship for normality is:

$$N = \frac{equivalent\ of\ solute}{liters\ of\ solute} = \frac{equiv}{V_L} = \frac{m}{eqw \times V_L}$$

Where:

m = the mass of the solute in grams eqw = the equivalent weight (grams / equivalent)  $V_1$  = volume of the solution in liters

Because equivalent weight and molecular weight are related, normality and molarity are related. In general:

$$Equivalent\ weight = \frac{Molecular\ weight}{a}$$

Where:

a = number of moles of acidic hydrogen (H<sup>+</sup>) per mole of acid that react, or the moles of OH<sup>-</sup> per mole of base that react. Thus the normality of a solution and its molarity are related:

$$N = a \times M$$

It should be obvious that a 1M HCl is also 1N, because 1 gram-equivalent weight of HCl is exactly 1 mol of HCl.

However, a 1M H<sub>2</sub>SO<sub>4</sub> solution is 2 N because 1 mol of H<sub>2</sub>SO<sub>4</sub> is equal to 2 equivalents of H<sub>2</sub>SO<sub>4</sub>.

In this experiment your solution of NaOH will be standardized by titrating it against a very pure sample of potassium acid phthalate,  $KHC_8H_4O_4$ , of known weight. Potassium acid phthalate (thereafter abbreviated as KHP) has only one replaceable acid hydrogen. Hence, it a monoprotic acid and has a molecular weight of 204.2g; hence 1 equiv of KHP weighs 204.2 g.

In the titration of a base against KHP, an equal number of equivalents of base and acid are present at the end point (more properly called the equivalence point). In other words, at the equivalence point:

 $Equivalents\ NaOH = Equivalents\ KHP$ 

Since:

Equivalents NaOH = 
$$N \times V_L = \frac{m}{eqw}$$

So that:

$$N_{base} \times V_{base} = \frac{m \ of \ KHP}{eqw \ of \ KHP}$$

If one measures the volume (in liters) of base required to neutralize a known weight (in grams) of KHP, it is possible to calculate the normality (N) of the base because the equivalent weight of KHP is known.

#### PROCEDURE:

- 1. Preparation of approximately 0.100 N of a sodium hydroxide solution. Measure 8.4 mL of 6 N sodium hydroxide solution in a 10 mL graduated cylinder. Pour the solution into a clean 500-mL plastic bottle. Dilute this solution to approximately 500 mL with distilled water. The volume of the sodium hydroxide solution does not have to be known accurately. This solution will be titrated in the next part of the experiment against KHP to determine its exact normality. Stopper firmly the plastic bottle. Mix the solution completely by inverting the plastic bottle ten times. Insufficient mixing of solutions is a common source of error in titration.
- 2. Standardization of the Sodium hydroxide solution. Prepare about 400-450 ml of CO2 free water by boiling for about 5 minutes the deionized water. Weigh from a weighing bottle triplicate samples of between 0.4 and 0.6 g each of pure potassium acid phtalate into 250ml Erlenmeyer flasks; accurately weigh to the nearest 0.000 1 g (Do not weigh the flask). Record the weights and label the three flasks in order to distinguish among them. Add to each sample about 100 mL of deionized water that has been freed from carbon dioxide by boiling, and warm gently with swirling until the salt is completely dissolved. Add to each flask two drops of phenolphthalein indicator solution. Rinse the previously cleaned buret with at least four 5-mL portions of the approximately 0.100 N of the sodium hydroxide solution that you have prepared. Discard each portion. Completely fill the buret with the solution and remove the air from the tip by running out some of the liquid into an empty beaker. Make sure that the lower part of the meniscus is at zero mark or slightly lower. Allow the buret to stand for at least 30 seconds before reading the exact position of the meniscus. Remove any hanging drop from the buret tip by touching it to the side of the beaker used for the washings. Record the initial buret reading. Slowly add the NaOH solution to one of your flasks of KHP solution while gently stirring the contents of the flask. As the NaOH solution is added, a pink color appears where the drops of the base come in contact with the solution. The coloration disappears with swirling. As the end

point is approached, the color disappears more slowly, at which time the sodium hydroxide solution should be added drop by drop. The end point is reached when one drop of the sodium hydroxide solution turns the entire solution in the flask from colorless to pink. The solution should remain pink when it is swirled. Allow the titrated solution to stand for at least 1 minute, so that the buret will drain properly. Remove any hanging drop from the buret tip by touching it to the side of the flask. Then wash it down, into the flask, with a stream of water from the wash bottle. Repeat this procedure with at least two more samples. From the data you obtain in at least three "good" titrations, calculate the normality of the sodium hydroxide solution as shown in the sample calculation given below.

#### **SAMPLE CALCULATIONS:**

What is the normality of a NaOH solution if 35.75 mL of it is required to neutralize 1.0700 g KHP?

$$N \times 0.03575L = \frac{1.070g}{204.2 \ g / equiv}$$

$$N = \frac{1.070g}{(204.2 \ g/equiv)(0.03573L)} = 0.1466N$$

| Experiment 33: Standard                   |                  | <u>IM Hydroxide So</u><br>) <u>RT FORM</u> | <u>lution with a Prim</u> | ary Standard |
|---|------------------|--|---------------------------|--------------|
| NAME:                                     | Date: _          | Partr                                      | ner(s):                   |              |
| Equivalent weight of Pota                 | ssium Acid Phta  | late: 204.2 g/eqı                          | uiv.                      |              |
| DATA:                                     |                  |  |                           |              |
|   | 1                | 2  | 3                         | 4            |
| Initial mass of KHP + bottle:             | g                | g  | g                         | g            |
| Final mass of KHP + bottle:               | g                | g  | g                         | g            |
| Mass of KHP used:                         | g                | g  | g                         | g            |
| Final buret reading:                      | mL               | mL   | mL                        | mL           |
| Initial buret reading:                    | mL               | mL   | mL                        | mL           |
| mL of NaOH used:                          | mL               | mL   | mL                        | mL           |
| CALCULATIONS:<br>Equivalents of KHP = Equ | uivalents of NaO | H = mass of KHI                            | P/equiv. wt of Kl         | НР           |
| Equiv of KHP (and NaOH):                  |                  |  |                           |              |
| Normality of NaOH = Equ                   | ivalents of NaOl | H/Liters of NaOH                           | 1                         |              |
| Normality of NaOH:                        | N                | N  | N                         | N            |
| Average Normality:                        |                  |  |                           | N            |
| Deviation $ d_i $ :                       | N                | N  | N                         | N            |
| Mean Deviation, d:                        |                  |  |                           | N            |
| Relative Mean Deviation                   |                  |  |                           | N            |
|   |                  |  |                           |              |



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|               |  |  |
|               |  |  |
|               |  |  |
|               |  |  |

# **APPENDIX I – Electronegativity of The Elements**

**Pauling's Electronegativity Values of the Elements** 

| Name        | Electronegativity | Name         | Electronegativity |              | Electronegativity |
|-------------|-------------------|--------------|-------------------|--------------|-------------------|
|             | Value             |              | Value             |              | Value             |
| Actinium    | 1.1               | Gold         | 2.5               | Promethium   |                   |
| Aluminum    | 1.5               | Hafnium      | 1.3               | Protactinium | 1.5               |
| Americium   | 1.3               | Holmium      |                   | Radium       | 0.9               |
| Antimony    | 1.9               | Hydrogen     | 2.1               | Radon        |                   |
| Argon       |                   | Indium       | 1.7               | Rhenium      | 1.9               |
| Arsenic     | 2.0               | lodine       | 2.5               | Rhodium      | 2.3               |
| Astatine    | 2.2               | Iridium      | 2.2               | Rubidium     | 0.8               |
| Barium      | 0.9               | Iron         | 1.8               | Ruthenium    | 2.2               |
| Berkelium   | 1.3               | Krypton      | 3.0               | Samarium     | 1.2               |
| Beryllium   | 1.5               | Lanthanum    | 1.1               | Scandium     | 1.4               |
| Bismuth     | 1.9               | Lawrencium   |                   | Selenium     | 2.4               |
| Boron       | 2.0               | Lead         | 1.8               | Silicon      | 1.8               |
| Bromine     | 2.8               | Lithium      | 1.0               | Silver       | 1.9               |
| Cadmium     | 1.7               | Lutetium     | 1.3               | Sodium       | 0.9               |
| Calcium     | 1.0               | Magnesium    | 1.2               | Strontium    | 1.0               |
| Californium | 1.3               | Manganese    | 1.6               | Sulphur      | 2.5               |
| Carbon      | 2.5               | Mendelevium  | 1.3               | Tantalum     | 1.5               |
| Cerium      | 1.1               | Mercury      | 2.0               | Technecium   | 1.9               |
| Cesium      | 0.7               | Molybdenum   | 2.3               | Tellurium    | 2.1               |
| Chlorine    | 3.0               | Neon         |                   | Terbium      |                   |
| Chromium    | 1.7               | Neptunium    | 1.3               | Thallium     | 1.8               |
| Cobalt      | 1.9               | Nickel       | 1.9               | Thorium      | 1.3               |
| Copper      | 2.0               | Niobium      | 1.6               | Thulium      | 1.3               |
| Curium      | 1.3               | Nitrogen     | 3.0               | Tin          | 1.8               |
| Dysprosium  | 1.2               | Nobelium     | 1.3               | Titanium     | 1.5               |
| Einsteinium | 1.3               | Osmium       | 2.2               | Tungsten     | 2.4               |
| Erbium      | 1.2               | Oxygen       | 3.5               | Uranium      | 1.7               |
| Europium    |                   | Palladium    | 2.2               | Vanadium     | 1.6               |
| Fermium     | 1.3               | Phosphorus   | 2.1               | Xenon        | 2.6               |
| Fluorine    | 4.0               | Platinum     | 2.3               | Ytterbium    |                   |
| Francium    | 0.7               | Plutonium    | 1.3               | Yttrium      | 1.2               |
| Gadolinium  | 1.2               | Polonium     | 2.0               | Zinc         | 1.7               |
| Gallium     | 1.6               | Potassium    | 0.8               | Zirconium    | 1.3               |
| Germanium   | 1.8               | Praseodymium | 1.1               |              |                   |

Percent Ionic Character of a Single Chemical Bond

|                                 | 0.1 |    |    |    |    |    |    |    |    |    |    |    |    |    |    |     |
|---------------------------------|-----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|-----|
| Percent ionic character %       | 0.5 | 1  | 2  | 4  | 6  | 9  | 12 | 15 | 19 | 22 | 26 | 30 | 34 | 39 | 43 | 47  |
|                                 |     |    |    |    |    |    |    |    |    |    |    |    |    |    |    |     |
| Difference in electronegativity |     |    |    |    |    |    |    |    |    |    |    |    |    |    |    | 3.2 |
| Percent ionic character %       | 51  | 55 | 59 | 63 | 67 | 70 | 74 | 77 | 79 | 82 | 84 | 88 | 89 | 91 | 92 | 95  |

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|-----------|---|----|---|
|-----------|---|----|---|

# **APPENDIX II – The Periodic Table**

# The Periodic Table of the Elements

|                      | ı                    |                       |                    |                      |                     |                        |                  |                     |                     |                   |                   |                       |                    |                         |                   |                     |                  |
|----------------------|----------------------|-----------------------|--------------------|----------------------|---------------------|------------------------|------------------|---------------------|---------------------|-------------------|-------------------|-----------------------|--------------------|-------------------------|-------------------|---------------------|------------------|
| 1                    |                      |                       |                    |                      |                     |                        |                  |                     |                     |                   |                   |                       |                    |                         |                   |                     | 2                |
| H                    |                      |                       |                    |                      |                     |                        |                  |                     |                     |                   |                   |                       |                    |                         |                   |                     | He               |
| Hydrogen<br>1,00794  |                      |                       |                    |                      |                     |                        |                  |                     |                     |                   |                   |                       |                    |                         |                   |                     | Helium<br>4.003  |
| 3                    | 4                    |                       |                    |                      |                     |                        |                  |                     |                     |                   |                   | 5                     | 6                  | 7                       | 8                 | 9                   | 10               |
| Li                   | Be                   |                       |                    |                      |                     |                        |                  |                     |                     |                   |                   | В                     | Č                  | Ň                       | ŏ                 | F                   | Ne               |
| Lithium              | Beryllium            |                       |                    |                      |                     |                        |                  |                     |                     |                   |                   | Boron                 | Carbon             | Nitrogen                | Oxygen            | ■'<br>Fluorine      | Neon             |
| 6.941                | 9.012182             |                       |                    |                      |                     |                        |                  |                     |                     |                   |                   | 10.811                | 12.0107            | 14.00674                | 15.9994           | 18.9984032          | 20.1797          |
| 11                   | 12                   |                       |                    |                      |                     |                        |                  |                     |                     |                   |                   | 13                    | 14                 | 15                      | 16                | 17                  | 18               |
| Na                   | Mg                   |                       |                    |                      |                     |                        |                  |                     |                     |                   |                   | Al                    | Si                 | P                       | $\mathbf{S}$      | Cl                  | Ar               |
| Sodium<br>22,989770  | Magnesium<br>24,3050 |                       |                    |                      |                     |                        |                  |                     |                     |                   |                   | Aluminum<br>26.981538 | Silicon<br>28.0855 | Phosphorus<br>30.973761 | Sulfur<br>32,066  | Chlorine<br>35,4527 | Argon<br>39,948  |
| 19                   | 20                   | 21                    | 22                 | 23                   | 24                  | 25                     | 26               | 27                  | 28                  | 29                | 30                | 31                    | 32                 | 33                      | 34                | 35.4527             | 36               |
|                      | 7-15000              |                       |                    |                      | 1000000             | 20000000               | 197900           | 10000001            |                     |                   | 1000000           |                       |                    |                         |                   | 2000000             |                  |
| K                    | Ca                   | Sc                    | Ti                 | $\mathbf{V}$         | Cr                  | Mn                     | Fe               | Co                  | Ni                  | Cu                | Zn                | Ga                    | Ge                 | As                      | Se                | Br                  | Kr               |
| Potassium<br>39.0983 | Calcium<br>40.078    | Scandium<br>44.955910 | Titanium<br>47.867 | Vanadium<br>50.9415  | Chromium<br>51.9961 | Manganese<br>54.938049 | 1ron<br>55.845   | Cobalt<br>58.933200 | Nickel<br>58.6934   | Copper<br>63.546  | Zinc<br>65.39     | Gallium<br>69.723     | Germanium<br>72.61 | Arsenic<br>74.92160     | Selenium<br>78.96 | 79.904              | Krypton<br>83.80 |
| 37                   | 38                   | 39                    | 40                 | 41                   | 42                  | 43                     | 44               | 45                  | 46                  | 47                | 48                | 49                    | 50                 | 51                      | 52                | 53                  | 54               |
| Rb                   | Sr                   | $\mathbf{Y}$          | Zr                 | Nb                   | Mo                  | Tc                     | Ru               | Rh                  | Pd                  | Ag                | Cd                | In                    | Sn                 | Sb                      | Te                | I                   | Xe               |
| Rubidium             | Strontium            | Yttrium               | Zirconium          | Niobium              | Molybdenum          | Technetium             | Ruthenium        | Rhodium             | Palladium           | Silver            | Cadmium           | Indium                | Tin                | Antimony                | Tellurium         | Iodine              | Xenon            |
| 85.4678              | 87.62                | 88.90585              | 91.224             | 92.90638             | 95.94               | (98)                   | 101.07           | 102.90550           | 106.42              | 107.8682          | 112,411           | 114.818               | 118.710            | 121.760                 | 127.60            | 126.90447           | 131.29           |
| 55                   | 56                   | 57                    | 72                 | 73                   | 74                  | 75                     | 76               | 77                  | 78                  | 79                | 80                | 81                    | 82                 | 83                      | 84                | 85                  | 86               |
| Cs                   | Ba                   | La                    | Hf                 | Ta                   | W                   | Re                     | Os               | Ir                  | Pt                  | Au                | Hg                | Tl                    | Pb                 | Bi                      | Po                | At                  | Rn               |
| Cesium<br>132,90545  | Barium<br>137.327    | Lanthanum<br>138,9055 | Hafnium<br>178.49  | Tantalum<br>180.9479 | Tungsten<br>183.84  | Rhenium<br>186,207     | Osmium<br>190.23 | 192.217             | Platinum<br>195.078 | Gold<br>196,96655 | Mercury<br>200.59 | Thallium<br>204,3833  | Lead<br>207.2      | Bismuth<br>208.98038    | Polonium<br>(209) | Astatine<br>(210)   | (222)            |
| 87                   | 88                   | 89                    | 104                | 105                  | 106                 | 107                    | 108              | 109                 | 110                 | 111               | 112               | 113                   | 114                |                         |                   | X                   |                  |
| Fr                   | Ra                   | Ac                    | Rf                 | Db                   | Sg                  | Bh                     | Hs               | Mt                  |                     | ,,,,              |                   |                       |                    |                         |                   |                     |                  |
| Francium             | Radium               | Actinium              | Rutherfordium      | Dubnium              | Seaborgium          | Bohrium                | Hassium          | Meitnerium          |                     | 9000000000        |                   |                       |                    |                         |                   |                     |                  |
| (223)                | (226)                | (227)                 | (261)              | (262)                | (263)               | (262)                  | (265)            | (266)               | (269)               | (272)             | (277)             | × ×                   |                    |                         | o.                |                     |                  |

| 58       | 59           | 60        | 61         | 62        | 63        | 64         | 65        | 66          | 67          | 68      | 69          | 70        | 71         |
|----------|--------------|-----------|------------|-----------|-----------|------------|-----------|-------------|-------------|---------|-------------|-----------|------------|
| Ce       | Pr           | Nd        | Pm         | Sm        | Eu        | Gd         | Tb        | Dy          | Ho          | Er      | Tm          | Yb        | Lu         |
| Cerium   | Praseodymium | Neodymium | Promethium | Samarium  | Europium  | Gadolinium | Terbium   | Dysprosium  | Holmium     | Erbium  | Thulium     | Ytterbium | Lutetium   |
| 140.116  | 140.90765    | 144.24    | (145)      | 150.36    | 151.964   | 157.25     | 158.92534 | 162.50      | 164.93032   | 167.26  | 168.93421   | 173.04    | 174.967    |
| 90       | 91           | 92        | 93         | 94        | 95        | 96         | 97        | 98          | 99          | 100     | 101         | 102       | 103        |
| Th       | Pa           | U         | Np         | Pu        | Am        | Cm         | Bk        | Cf          | Es          | Fm      | Md          | No        | Lr         |
| Thorium  | Protactinium | Uranium   | Neptunium  | Plutonium | Americium | Curium     | Berkelium | Californium | Einsteinium | Fermium | Mendelevium | Nobelium  | Lawrencium |
| 232.0381 | 231.03588    | 238.0289  | (237)      | (244)     | (243)     | (247)      | (247)     | (251)       | (252)       | (257)   | (258)       | (259)     | (262)      |