NAME: HONORS CHEMISTRY

SECTION: Determining an Empirical Formula

An ionic compound is composed of ions – atoms or groups of atoms that have a positive or negative charge. Oppositely charged ions arrange themselves into an extended, three-dimensional structure called a crystal lattice. The net attractive forces among oppositely charged ions in the crystal structure are called ionic bonds. Although composed of charged ions, ionic compounds are electrically neutral. The ratio of oppositely charged ions in the crystal structure is such that the positive charge contributed by the cations in equal to or balanced by the negative charge contributed by the anions. There is no net or overall charge on an ionic compound.

The *empirical formula* of an ionic compound indicates the smallest whole number ratio of each type of ion in the crystal structure and is called a formula unit. For example, magnesium chloride has the empirical formula $MgCl_2$. Magnesium cations (Mg^{2+}) and chloride anions (Cl^-) combine in a 1:2 ratio to form the $MgCl_2$ formula unit. The overall charge on ionic compounds is always zero.

Many ionic compounds can be prepared in the lab using *precipitation reactions*. When solutions of two ionic compounds are combined, the ions may rearrange to form a new ionic compound that is insoluble in water. An example of this type of reaction is the formation of solid barium sulfate when barium chloride and sodium sulfate are combined in solution (Equation 1a). In Equation 1b, only the ions that form the precipitate are represented. This makes it easier to recognize what happens in the precipitation reaction.

$$BaCl_2(aq) + Na_2SO_4(aq) \rightarrow BaSO_4(s) + 2NaCl(aq) \qquad \textit{Equation 1a}$$

$$Ba^{2+}(aq) + SO_4^{2-}(aq) \rightarrow BaSO_4(s) \qquad \textit{Equation 1b}$$

According to the balanced equation for this reaction, barium ions (Ba^{2+}) combine with sulfate ions (SO_4^{2-}) in a 1:1 ratio to form barium sulfate ($BaSO_4$). This ratio can be observed experimentally in the lab by mixing $BaCl_2$(aq) and Na_2SO_4(aq) solutions containing equal amounts (concentrations) of barium and sulfate ions, respectively. The maximum amount of precipitate will be obtained when equal volumes (a 1:1 ratio) of the two solutions are combined. A similar approach can also be used to determine the formula of an unknown ionic compound.

The purpose of this experiment is to determine the empirical formula of an unknown ionic compound. Two solutions containing equal amounts (concentrations) of two reactant ions will be combined in a series of reactions. However, the charge of the metal cation is not known. In each reaction, the totally volume of the two solutions will be held constant while the volume ratio of the reactants is varied. The amount of precipitate obtained in each reaction will be measured and plotted against the volume ratio to find the empirical formula of the product.

Materials

- 0.1 M copper chloride solution
- Micro-stirring rod
- 0.1 M sodium phosphate solution, Na_3PO_4,
- Marking pen or wax pencil
- Metric ruler, marked in mm
- Pipets, Beral-type, 2
- 96-well reaction plate
- Test tubes, small, 7

Roles

Project Manager ______________________________

Materials Manager ______________________________

Quality Control Manager ______________________________

Pre-Lab Questions

Answer these questions on a separate sheet of paper and turn them in the day of the lab.

1. Many common drugstore chemicals are ionic compounds. Write the correct empirical formula for each of the following compounds.

Common name:	Milk of magnesia	Washing soda	Epsom salt
Chemical name:	Magnesium hydroxide	Sodium carbonate	Magnesium sulfate

2. Solutions of iron(III) chloride and sodium hydroxide were mixed in a series of precipitation reactions, as described in this experiment.

   What volume ratio of reactants gave the most precipitate (see Table 1)?. Explain.

Table 1.

Test tube	**1**	**2**	**3**	**4**	**5**	**6**	**7**
$FeCl_3$, 0.1 M, mL	5	10	12	15	17	20	24
NaOH, 0.1 M, mL	55	50	48	45	43	40	36
Volume of precipitate, mL	1	10	14	20	4	1	0

Safety Precautions

Copper chloride and sodium phosphate solutions are skin and eye irritant; additionally, they are slightly toxic by ingestion. Avoid contact of all chemicals with eyes and skin. Wear chemical splash goggles and chemical-resistant gloves and apron. Wash hands thoroughly with soap and water before leaving the lab.

Hypothesis: Copper typically forms either a +1 cation or a +2 cation. Based on your knowledge of chemical formulas, predict the ratio of the ions in the compound formed between each copper ion and the phosphate ion. As a group, use the charges on the ions to predict the empirical formula of each possible product. ***Be sure to include this hypothesis, using an "If…then…" format in the introduction to your lab report.***

Procedure

1. Label seven micro test tubes #1-7 with a marking pen and place them in a test tube rack or in a 96 well-reaction plate.
2. Obtain two micropipets: one filled with 0.1M copper chloride solution and another filled with 0.1M sodium phosphate. Record the color of each solution in the data table.
3. Carefully add the appropriate number of drops of copper chloride solution to each test tube #1-7, as shown in Table 2. *Note:* Exact volumes are very important – hold the pipet vertically to obtain uniform size drops.
4. Carefully add the appropriate number of drops of sodium phosphate solution to each test tube, as shown in Table 2.

Table 2.

Test Tube	**1**	**2**	**3**	**4**	**5**	**6**	**7**
Copper solution, 0.1M drops	9	8	6	5	4	2	1
Na_3PO_4, 0.1M drops	1	2	4	5	6	8	9

5. Use a *clean* micro-stirring rod to stir each reaction mixture in test tubes #1-7. Let the tubes sit undisturbed for 10-15 minutes to allow the precipitates to settle.
6. During this time, determine the volume (drop) ratio of copper chloride and sodium phosphate solutions in each test tube. Write this ratio in the data table. *Example:* In test tube #1, 3 drops of copper chloride solution and 27 drops of Na_3PO_4 correspond to a 1:9 ratio.
7. After the precipitates have settled, observe the appearance of the products (*both the solid and the solution*). Record the observation in the data table in the space provided. Be as detailed as possible.
8. Use a metric ruler to measure the height of the precipitate of millimeters in each test tube. Read from the top of the solid material to the bottom center of the test tube. Record each height in mm in the data table.
9. Dispose of the contents of the test tubes as directed by your instructor.

Data Table

Color of copper-chloride solution		**Color of Na_3PO_4 Solution**	
Appearance of precipitate			

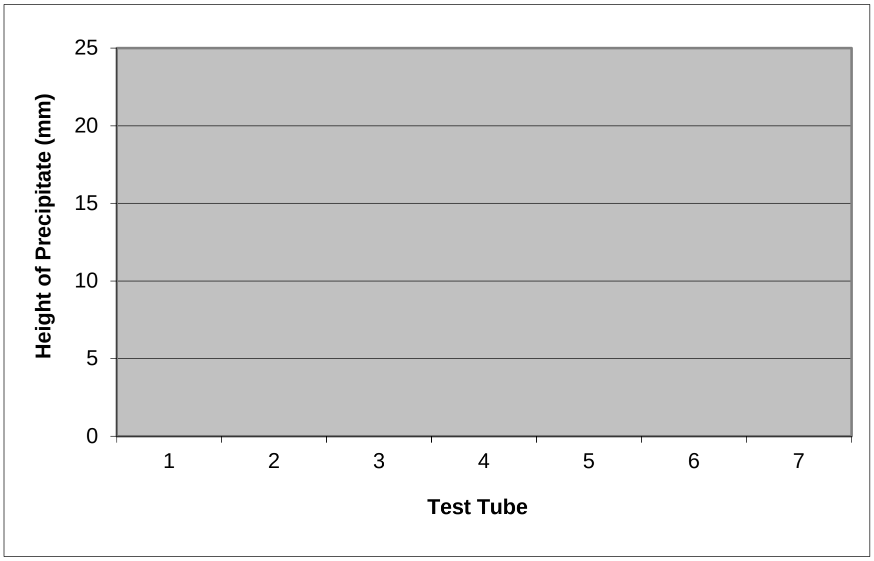
Precipitation Reactions

Test tube	1	2	3	4	5	6	7
Volume Ratio * (Drops of copper chloride solution:Drops Na_3PO_4 solution							
Height of Precipitate (mm)							
Observations Color of supernatent							

*Reduce the volume ratio to the simplest whole-number ratio

Analyze and Apply Answer these questions in complete sentences.

1. Complete the following bar graph to show the height of the precipitate in each test tube.



2. Which test tube had the greatest amount of precipitate?

3. Based on your data, was the Cu^{+} or the Cu^{2+} cation present? Explain.

4. Based on your data, write the formula and give the name of the precipitate formed.

5. (a) Which test tubes showed evidence of unreacted copper ions in the supernatant when the reaction was complete? Explain why unreacted copper ions were present in these tubes based on the volume ratio of the solutions used.

   (b) How could you tell that all of the copper ions had reacted in a particular test tube? Which test tubes showed such evidence? Explain, based on the volume ratio of solutions used.

6. What was the total number of drops of solution in each test tube? Why was it necessary to keep the total volume of reactant constant in each test tube?

7. Does the *height* of precipitate in each test tube accurately reflect the *amount* of precipitate in each case? *Hint:* Compare the shape of a test tube to that of a graduated cylinder. What effect does this error have on the conclusions reached in this experiment?

In your conclusion, compare your experimental formula to the ratios you predicted in the hypothesis. Discuss sources of experimental error and ways to improve this experiment.