The Reaction of Calcium Chloride with Carbonate Salts

PRE-LAB ASSIGNMENT:

Reading: Chapter 3 & Chapter 4, sections 1-3 in Brown, LeMay, Bursten, & Murphy.

1. What product(s) might be expected to form when solid lithium carbonate is added to an aqueous solution of calcium chloride? Write a balanced chemical equation for this process.
2. How many grams of lithium carbonate would you need to fully react with one mole of calcium chloride? Please show your work.

INTRODUCTION: The purpose of this lab is to help you discover the relationships between the reactants and products in a precipitation reaction. In this lab you will react a calcium chloride solution with lithium carbonate, sodium carbonate, or potassium carbonate. The precipitate that results will be filtered and weighed. In each determination you will use the same amount of calcium chloride and different amounts of your carbonate salt. This experiment is a "discovery"- type experiment. The procedure will be carefully described, but the analysis of the data is left purposely vague. You will work in small groups to decide how best to work up the data. In the process you will have the chance to discover some principles, to use what you have learned in lecture, and to practice thinking about manipulative details and theory at the same time. Plotting your data in an appropriate manner should verify the identity of the precipitate and clarify the relationship between the amount of carbonate salt and the yield of precipitate.

Predicting the formulas of ionic compounds. Compounds like calcium chloride ($CaCl_2$) and sodium carbonate (Na_2CO_3) are ionic substances. Soluble ionic substances dissociate in aqueous solution to form ions as shown below for calcium chloride.

$$CaCl_2\,(aq) \rightarrow Ca^{2+}\,(aq) + 2\,Cl^-\,(aq)$$

A list of common ions is given in your text. A short excerpt is tabulated below. The charges on the ions can be used to predict the formulas of ionic compounds as the ions combine to give electrically neutral compounds. For example, the combination of K^+ and PO_4^{3-} would give K_3PO_4, which has a net charge of zero. If more than one polyatomic ion such as nitrate ion is needed to balance the charge, it is enclosed in parentheses with the number of times it occurs indicated with a subscript to the right of the parentheses, for example, in calcium nitrate, $Ca(NO_3)_2$.

Some Common Ions

Cations		Anions	
H^+	hydrogen ion	OH^-	hydroxide ion
Na^+	sodium ion	Cl^-	chloride ion
K^+	potassium ion	CO_3^{2-}	carbonate ion
Ca^{2+}	calcium ion	NO_3^-	nitrate ion
Ag^+	silver ion	PO_4^{3-}	phosphate ion
NH_4^+	ammonium ion	SO_4^{2-}	sulfate ion

Predicting the product of precipitation reactions. Sometimes a precipitate results when two soluble ionic compounds are mixed in aqueous solution. For example, a precipitate is formed when solutions of silver nitrate and sodium chloride are mixed. How would you predict the identity of the precipitate? This can be done in two steps. First, break the parent compounds into their respective ions, and then exchange the ions between partners to predict the products. For example, silver nitrate and sodium chloride dissociate into ions in solution:

$$AgNO_3\,(aq) \rightarrow Ag^+\,(aq) + NO_3^-\,(aq)$$

$$NaCl\,(aq) \rightarrow Na^+\,(aq) + Cl^-\,(aq)$$

In the mixture of silver nitrate and sodium chloride solutions, we consider the products formed by the exchange of partners (each cation ends up with a different anion than it started with), predicting the possible products AgCl and $NaNO_3$. Therefore, the observed precipitate could be AgCl or $NaNO_3$, or it could be both. Later in this course you will learn how to predict which ionic substances will be insoluble. In this example, AgCl is insoluble in water and precipitates out of solution, while $NaNO_3$ is soluble, so the Na^+ and NO_3^- ions remain dissolved and, therefore, uncombined in solution. This reaction would then have the balanced equation:

$$AgNO_3\,(aq) + NaCl\,(aq) \rightarrow AgCl\,(s) + Na^+(aq) + NO_3^-(aq)$$

The net ionic equation for this reaction is:

$$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$$

The general rules for water solubility of common ionic compounds include the following two rules, with rule one taking precedence over rule two:

1. All common compounds of the alkali metals and the ammonium ion are soluble.
2. Almost all carbonate, phosphate, and hydroxide compounds are insoluble.

Therefore, $MgCO_3$, Ag_3PO_4, and $Ca(OH)_2$ are predicted to be insoluble in water, but Na_2CO_3, $(NH_4)_3PO_4$, and KOH are predicted to be soluble.

PROCEDURE: You will work individually on the experimental portion of this laboratory to analyze two different samples that are assigned to you, and then you will share your data with your assigned group. At the end of the lab, the data from the entire class will be combined and emailed to you. For each sample you will react the same amount of calcium chloride solution with different (assigned) amounts of your carbonate salt. Your instructor will tell you which carbonate salt (lithium, sodium, or potassium carbonate) to work with and what weight range you are to use.

1. Label two clean and **DRY** Gooch crucibles. Weigh these crucibles to an accuracy of 0.5 mg (i.e. 0.0005 g). This step can be completed at any time before step 8, so plan your work to use time efficiently. The accuracy of this experiment will not be compromised by the use of your fingers to handle the crucibles.

2. Label two clean, DRY weighing bottles and use them to weigh out two samples of your carbonate salt within the mass ranges assigned to you. Record your weights to an accuracy of 0.5 mg. For example, if your instructor assigns the weight range of 0.30 to 0.45 g for one of your samples, any weight within this range is fine, but you must know that weight to 0.5 mg. Thus, 0.4255 g would be within the 0.30 to 0.45g range and to the required accuracy.

3. Quantitatively transfer your carbonate salts to two separate clean, labeled 150 mL beakers, making sure you record which sample goes into which beaker. To transfer the salt, empty the contents of the weighing bottle carefully into the beaker. Measure 50 mL of 0.05 M ammonia solution for each salt into a graduated cylinder. Use small portions of this ammonia solution to rinse each weighing bottle, adding these rinses to the proper beaker, using all 50 mL of the ammonia solution. Ammonia is used instead of distilled water to make your solution slightly basic. A basic solution will minimize any loss of the precipitate you will be creating in this experiment. Stir the solution with a glass-stirring rod until all of the solid dissolves. Do not remove the stirring rod once you have placed it into the beaker; you do not want to lose any of the solution. If you have difficulty dissolving the salt, there is an ultrasonic bath available for your use. Simply place the beaker into the bath for a few minutes until dissolution is complete. (The beaker should not touch the walls or bottom of the ultrasonic bath.)

4. Heat the beakers on hot plates until water vapor begins to condense on the sides of the beaker. Do not allow the solution to boil.

5. Carefully dispense 10.00 mL of the $CaCl_2$ solution directly into each beaker. The $CaCl_2$ solution is in a dispenser bottle that is set to deliver this precise volume of solution. The concentration of $CaCl_2$ will be approximately 22.2g/L, but the exact concentration will be on the label. Be sure to write the exact concentration in your notebook.

6. Stir each solution occasionally while continuing to heat it for two minutes

7. Allow the beakers to cool until they are at room temperature. While waiting label two clean and **DRY** Gooch crucibles. Weigh each Gooch to an accuracy of 0.5 mg (i.e. 0.0005 g). If your two beakers are still hot, cooling can be hastened by placing the beakers into an ice-water bath for a few minutes. Upon mixing does the entire beaker feel cool to the touch? If so, then you are ready to continue.

8. Pour each mixture carefully into one of the pre-weighed Gooch crucibles to filter it. The filtration apparatus is shown in Figure 1. When pouring from the sample beaker, pour down the stirring rod to avoid spilling, as shown in Figure 2.

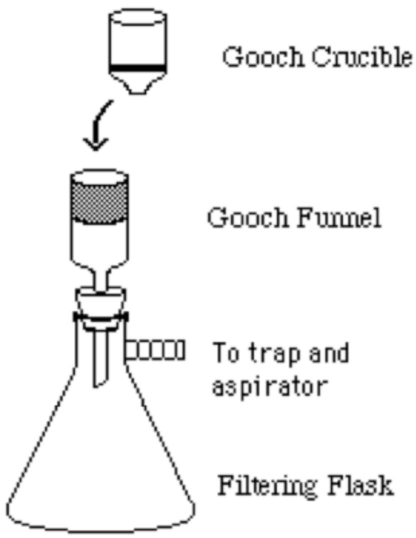

Figure 1. Filtration apparatus

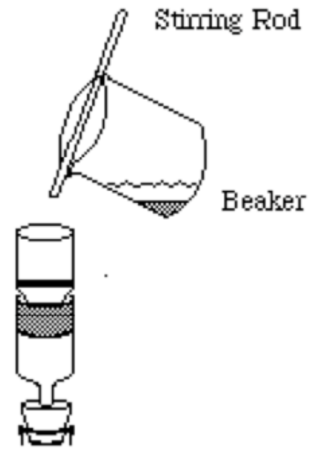

Figure 2. Pouring down a stirring rod.

9. Use a wash bottle containing 0.05 M ammonia solution to rinse the precipitate into the Gooch crucible, as shown in Figure 3. Rinse the sample beaker at least three times to ensure complete transfer. Do not dump out the liquid in your filter flask, as it will be needed later.

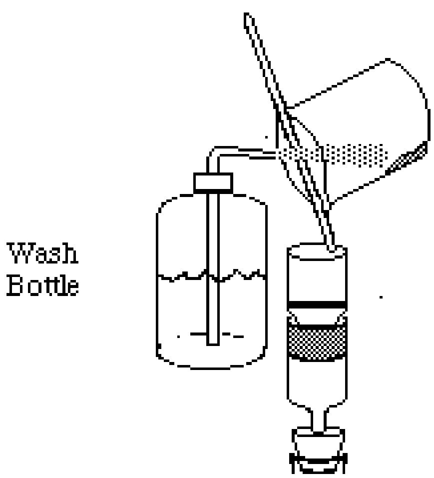

Figure 3. Washing the precipitate into the Gooch crucible.

10. Place the Gooch crucibles *on their sides* in a Petri dish. Record the number labeled on your Petri dish into your lab notebook. Place the Petri dish in the oven at 150-160 °C for 30 minutes. While you wait for the samples to dry, set up an EXCEL spreadsheet to accept your data for plotting. This is the time for talking with your group to figure out what you are going to do with your data.

11. Remove the Petri dish from the oven (using your lab towel) and allow the Gooches to cool to room temperature. Remember that hot objects will appear lighter than they should on an analytical balance because

they create air currents that lift the balance pan. Weigh the crucibles to find the mass of resulting precipitate for each.

12. Procedure for cleaning the Gooch **after** your group graph(s) is made and checked by your instructor:
    a) Leave the suction filtration apparatus set up with the ammonia solution still in the bottom of the flask.
    b) Carefully scrape as much of the precipitate as possible from the Gooch into the filter flask.
    c) Wash the Gooch at a sink using warm water and a brush to remove more of the solids.
    d) Return the Gooch to the suction filtration apparatus and carefully pour in some 3 M HCl with the suction off. After the fizzing stops, attach the hose and remove the solution by suction. Repeat.
    e) Use the suction to pull several portions of distilled water through the Gooch. Set the cleaned Gooch into the Petri dish and leave it on top of your fume hood. Be sure to leave your assigned index card with the Petri dish, too.

CALCULATIONS: Share your data with your assigned group. Make sure each member of your group used the same carbonate salt. Your group is on its own, except for the following hints, comments, and questions.

First, predict what the precipitate is and balance the chemical equation. What relationships between the reactant and product do you expect? Try to come up with a way of plotting the data that makes the underlying relationships as clear as possible. NOTE: In making a plot, the variable you control is plotted on the horizontal axis and the experimental result is plotted on the vertical axis. Try drawing straight lines through your data, but don't try to fit a straight line to all the data points! **Why?**

Enter your initial salt mass and final precipitate mass into the class spreadsheet at the front table, for your two samples. Your instructor will send a copy of all the class data to you. At first the data from three carbonate salts may appear completely different. However, the way you analyze and plot this data can resolve these differences. Chemical theories are often judged on the basis of how well they unify and explain data from different experiments. See if your graphs can do that.

What should be in your laboratory notebook now?

- Keep track of all weight measurements in your lab notebook (record which balance you used) and record all observations. Remember to include units. For calculations, all members of the group should keep track of the various calculations and theories that were tried. Did you use any literature values? If so, include the reference for each.

- Identify your **group** precipitate and write a balanced chemical reaction, in both total ionic and net ionic forms, for your carbonate salt.

- Show your example calculations for converting from your experimental precipitate mass to the final values that you decided to plot on the group graph.

- If you used a spreadsheet for calculations, be sure to include one example for each type of calculation/formula used. Note that statistical functions (average, standard deviation, etc.) do not have to be detailed in this way.

What should be attached to your discussion now, but put into your lab notebook later?

- Attach a copy of your final graph for your group. Clearly indicate on this graph your two data points. Remember, each graph made should include a title, have two labeled axes, include applicable units, give trend-lines and corresponding R^2 value for each trendline.

- Attach a copy of any graph(s) you made in order to interpret the provided data from the class today. Write the two balanced chemical equations for the salts you did not personally analyze directly on the graph(s).

<u>What should be in your Discussion?</u>

On a single typed page address your objective. Explain what you graphed for the group data and the resulting shape of the graph. What is the significance of the slope(s) of the line(s)? What is the significance of the intersection of the two lines? How does your graph indicate what the precipitate is? Identify your precipitate. Write the balanced chemical reaction that enabled this precipitate to form. Compare and contrast the graph of the class data (all three salts) to your group graph. Is the precipitate the same for all groups? Comment on two possible sources of error. Give a concise explanation of how each of these errors may have directly impacted the final results (did they make your results too high, too low, etc., explain exactly how that worked). What assumption do you make about the purity of the isolated product?

Please place this sheet of paper, the group graph and the class graph (stapled together) into the white bookcase outside Keyes 310.

Acknowledgements: This laboratory was inspired by a similar experiment at Holy Cross, and J. M. DeMoura, J. A. Marcello, *J. Chem. Ed., 64* (5), 452 (1987).