Experiment 3: Reaction of Calcium Chloride with Carbonate Salts

Reading: Chapter sections 3.3, 3.4, 3.7 and 4.2 in your text, and this lab handout

Ongoing Learning Goals:

- To use a scientific notebook as a primary record of procedures, data, observations, and example calculations
- To make scientific measurements
- To present your formal results through a laboratory report along with proper citations
- To use Excel to tabulate, calculate, analyze, and graph scientific data
- To evaluate the uncertainty (error) in scientific measurements, and understand the causes of the underlying uncertainty

Additional Learning Goals for Experiment 3:

- To apply balanced chemical equations and stoichiometric relationships to quantitative measurements
- To gain experience conducting aqueous precipitation reactions, including the quantitative chemical transfer and isolation of solid products

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. 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.

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

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 (we know this because the salts dissolve):

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

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 with one carbonate salt assigned to you. You will then partner with other students to analyze the data for the group's carbonate salts. Step by step instructions follow.

1. Weigh your assigned sample of carbonate salt into a weighing bottle ensuring that the mass is within the assigned range. Record the weight (to an accuracy of 0.5 mg) and the balance number. 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.
2. Measure 50 mL of 0.05 M ammonia solution in a graduated cylinder. Carefully add the contents of the weighing bottle (step 1, above) into a clean 150 mL beaker, and use small portions of your ammonia solution to rinse the weighing bottle contents, adding these rinses to the beaker. Be sure to use all 50 mL of the ammonia solution. 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. 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.
3. Heat the beaker on a hot plate until water vapor begins to condense on the wall of the beaker. Avoid boiling the solution.
4. Carefully add 10.0 mL of the $CaCl_2$ solution into the beaker. Record the concentration of the $CaCl_2$ solution.
5. Heat and stir the contents of the beaker for two more minutes after adding the $CaCl_2$ solution. Let the beaker cool to room temperature using an ice-water bath as necessary.
6. While your beaker is cooling, carefully weigh a clean and **DRY** Gooch crucible to an accuracy of 0.5 mg (i.e. 0.0005 g). Use the same balance as Step 1 and record the mass.
7. Carefully filter the contents of your beaker through the pre-weighed Gooch crucible, as shown in Figures 1-2. The goal is to ensure that all the precipitate in your beaker is quantitatively transferred to the crucible. So, when pouring out of the beaker, use the stirring rod as shown in Figure 2 to minimize spillage and loss of precipitate.

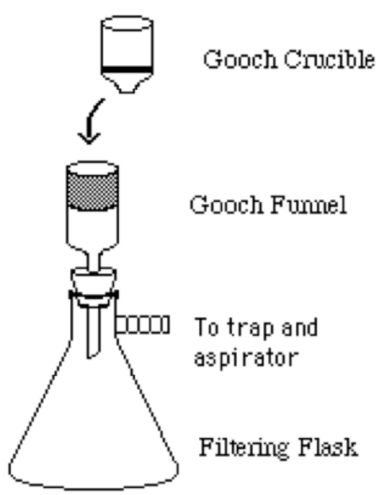

Figure 1: Filtration apparatus assembly

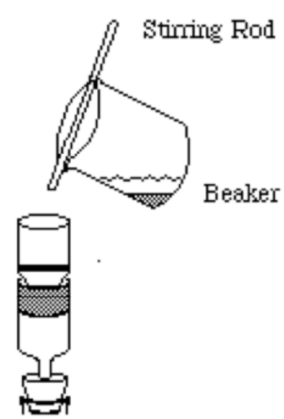

Figure 2: Pouring technique

8. Use a wash bottle containing cold 0.05 M ammonia solution to rinse the precipitate into the Gooch crucible. Rinse the beaker at least three times to ensure complete transfer.
9. Place the Gooch crucible in an assigned Petri dish and record the number of the dish in your notebook. Place the Petri dish with your crucible in an oven for at least 30 minutes. Record the oven temperature. **While you wait for the sample to dry, open the Google Sheet for data analysis with your partners. Please refer to instructions for data analysis that are given later in this handout.**
10. Remove the Petri dish and crucible from the oven and *allow it to cool to room temperature*. Weigh the crucible to 0.0005 g. Use the same balance from Step 1 and record the mass. Calculate the mass of your precipitate and record it in your notebook. You should add this data point to your group dataset and clearly identify it as yours with your initials.

Procedure for data analysis: (Please do the following while you wait for your crucible to dry in the oven)

1. Write a balanced equation for the chemical reaction between $CaCl_2$ and your carbonate salt. Identify the precipitate you made (your solid product) and recognize the quantitative relationship between the reactants and products.
2. Using the data shared in the class Google Sheet, plot the data (a "marked scatter" plot) with the product on the Y-axis and starting salt on the X-axis.
3. As a class, look at the scatter plot and decide how you might modify (transform) the experimental data to best represent the reaction stoichiometry. At this point it may be helpful to save the data to Excel to provide expanded graphing capabilities.
4. Your plot should have two regions separated by an inflection point. Do a separate linear fit to the data in each region (*yes, that means two separate trendlines*). Once all the data points have been included in your datafile, and you have added your trendlines, save the file to each group member's folder on the fileserver. Print your finalized plot and attach a copy in your notebook (you do not have to print/attach the data table).
5. Looking at all of the class data, make observations about the similarities/differences in the data obtained using the three different carbonates salts and record your observations in your notebook.

Procedure for cleaning the crucible after your graph is made and checked by the instructor:

1. Leave the suction filtration apparatus set up with the ammonia solution still in the bottom of the flask.
2. Return the crucibles 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 the HCl wash a second time.
3. Use the suction to pull several portions of deionized water through the crucibles.

What should be in your laboratory notebook (*in addition to title, purpose, procedure, observations, etc.*)

1. Keep track of all mass measurements in your lab notebook, identify the balance used, and record all observations. Always remember to include units.

2. Identify your precipitate and write a balanced chemical equation for the reaction of your carbonate salt.

3. Attach the final plot of the combined data from the class with trendlines and notation of the key sections of the plot.

Laboratory report: Use the **Report Form** for Experiment 3.

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