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GRAVIMETRIC ANALYSIS OF CALCIUM AND HARD WATER LAB

Introduction: In certain areas of the country, the presence of hard water poses significant problems in water supply systems. Various water softening techniques are used to remove the cations responsible for water hardness. This investigation involves the application of gravimetric analysis to test samples for the amount of water hardness and calcium ions.

Background:

Water from natural sources may contain a number of dissolved substances. The amount and nature of these dissolved substances varies depending on the geography of the area and the journey the water has taken. As water travels through the ground or over the surface of the land, it can dissolve naturally occurring minerals. As minerals dissolve in the water, the compounds separate into their respective cations and anions. Common cations in water include Na^{+}, Ca^{2+}, Mg^{2+}, and Fe^{3+}, while the principal anions in water are Cl^{-}, HCO_3^{-}, NO_3^{-}, and SO_4^{2-}. The main ions contributing to water hardness are Ca^{2+}, Mg^{2+}, and, to a lesser extent, Fe^{3+}. Their presence makes it difficult for soaps to lather and also causes a "scum" to form. Equation 1 (where R is a long hydrocarbon chain) shows the precipitation reaction between alkyl sulfate anions in a typical soap with calcium ions in hard water. The main problem due to water hardness in industrial pipes or boilers is the buildup of solid $CaCO_3$, which precipitates out and causes thick deposits to form in pipes and other appliances.

$$\underset{}{Ca^{2+}(aq)} + \underset{\textit{Soap}}{2\,ROSO_3^{-}(aq)} \rightarrow \underset{\textit{Soap scum}}{Ca(ROSO_3)_2(s)} \qquad \textit{Equation 1}$$

There are many different ways to "soften" water. One of the most common ways to remove ions is by ion exchange. The **ion exchange** process uses a resin to replace some of the ions that cause hardness with ions that do not. Hardness is commonly measured in units of grains per gallon or milligrams per liter (also known as parts per million), and is classified by the U.S. Department of the Interior and the Water Quality Association as follows:

Classification	**mg/L or ppm**	**grains/gal**
Soft	0 – 17.1	0 – 1
Slightly hard	12.7 – 60	1 – 3.5
Moderately hard	60 – 120	3.5 – 7.0
Hard	120 – 180	7.0 – 10.5
Very hard	180 and over	10.5 and over

Although several ions contribute to water hardness, the units of mg/L or ppm are defined in terms of the equivalent mass (milligrams) of $CaCO_3$ that would be present per liter of water. In this investigation, gravimetric analysis will be used to precipitate and isolate solid $CaCO_3$ from water samples and determine water hardness. Many municipal water treatment plants use soda ash (sodium carbonate, Na_2CO_3) and lime (calcium hydroxide, $Ca(OH)_2$) to chemically remove calcium and magnesium ions, respectively, from hard water.

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Experiment Overview:

The purpose of this advanced inquiry lab is to investigate the suitability of gravimetric analysis for determining the amount of water hardness in the form of calcium carbonate, $CaCO_3$, in various water samples. Six samples, representing a wide range of potential water hardness, from 50 ppm to 500 ppm, will be analyzed by various student groups as part of a cooperative class investigation to determine the accuracy and sensitivity of gravimetric analysis for water hardness testing. Note that all water samples have been concentrated by a factor of 100 for the purpose of quantitative analysis, in particular, quantitative transfer and vacuum filtration. The precipitation reaction involves preparing and combining solutions of Na_2CO_3 and $CaCl_2$. The balanced chemical equation for this reaction predicts the amount of precipitate that will be formed. Careful isolation, drying, and weighing of the precipitate will confirm the calculations and the percent yield. The procedure provides a model for guided-inquiry design of the cooperative class investigation described above.

Pre-Lab Questions: Answer these after the Purpose section of your lab write-up.

1) Define the term gravimetric analysis. Describe the procedure used in this activity, and identify two other common examples of gravimetric analysis. Cite your sources!
2) Write the balanced chemical equation for the reaction between calcium chloride and sodium carbonate.
3) Calculate the number of moles of each reactant in the Introductory Activity (see steps 1 and 2). Identify the limiting reactant in the reaction and determine the theoretical amount of $CaCO_3$ that should be produced.
4) As noted in the Background section, hardness levels are calculated by assuming that all the "hard" metal ions come from dissolved calcium carbonate and are reported in mg $CaCO_3$/L. Calculate the equivalent water hardness in mg $CaCO_3$/L for a calcium chloride solution containing 0.1 M Ca^{2+} ions.

Materials:

- Calcium chloride, anhydrous, 2 g
- Hard water samples, 20 mL
- Sodium carbonate solution, 0.5 M
- Sodium carbonate, anhydrous, 2 g
- Distilled water
- Electronic balance
- 100-mL beaker (3)
- 250-mL vacuum filter flask
- Buchner funnel
- Vacuum pump
- Filter paper disk (3)
- 100-mL graduated cylinder
- Spatula
- Watch glass (2)
- Weighing dish (2)

Safety Precautions:

Sodium carbonate is irritating to body tissues. Anhydrous calcium chloride is moderately toxic by ingestion and generates a great deal of heat when dissolved in water. Avoid contact of all chemicals with eyes and skin. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Wash hands thoroughly with soap and water before leaving the laboratory.

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Introductory Activity: This will be done as a demo. Include your data tables and calculations following your Pre-Lab Questions in your lab notebook.

Precipitation Reaction and Vacuum Filtration

1) Weigh 2.5 g of sodium carbonate and place in a clean, dry 100-mL beaker. Record the precise mass and dissolve the solid in 20 mL of distilled water.
2) Weigh 2.0 g of calcium chloride and place in a clean, dry 100-mL beaker. Record the precise mass and dissolve the solid in 20 mL of distilled water.
3) Combine the two solutions by slowly adding the sodium carbonate solution to the calcium chloride solution. Record all observations about the reaction.
4) Separately weigh a piece of filter paper and a watch glass and record their masses.
5) Set up a vacuum filtration apparatus.
6) Isolate the precipitate by vacuum filtration. Careful transfer techniques are essential for accurate results!
7) Place the watch glass and filter paper containing precipitate in a lab oven to dry at 100^{o}C for 10 – 20 minutes. Monitor and carefully break up the solid with a spatula to ensure complete drying.
8) Calculate the percent yield of calcium carbonate.

Guided-Inquiry Design and Procedure: Write the answers to these questions after your Introductory Activity Calculations. Sketch your procedure after these questions.

1) The ideal precipitate in a gravimetric analysis procedure should be insoluble and have a known composition. Look up the properties of calcium carbonate and discuss its advantages and possible disadvantages for gravimetric analysis of calcium. Cite your sources!
2) Based on solubility rules, what ions in water might interfere with the analysis of calcium ions by precipitation of calcium carbonate?
3) Precipitate particles in gravimetric analysis must be large enough to be collected by filtration—smaller particles may pass through or clog the filter. Discuss how the following techniques will help prevent product loss and ensure product purity in a gravimetric procedure.
   a) Add the precipitant slowly with vigorous mixing.
   b) "Digest" the precipitate by allowing it to stand in contact with the solution and/or heating the mixture for 10 – 15 minutes.
   c) Rinse the precipitate with a small amount of water after filtration.

Six water samples containing known concentrations of calcium chloride are available for analysis as part of a cooperative class activity. Each student group should analyze two different samples. The recommended sample volume for the precipitation reaction is 20 mL. Complete this table below in your lab notebook with the results of the calculations from Questions 4 – 6 below. You should include your calculations for ONLY Sample 2 for these questions.

Sample	**[$CaCl_2$] (M)**	**Moles of Ca^{2+} in 20 mL of Solution**	**Theoretical Amount of $CaCO_3$ Precipitate**	**Volume of 0.5 M Na_2CO_3 (20% excess)**	**Theoretical Water Hardness, mg/L**
1	0.400	0.00800	0.801 g	19.3 mL (20)	400. mg/L
2	0.200				
3	0.500				
4	0.100				
5	0.050				
6	0.300				

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4) Calculate the number of moles of Ca^{2+} ions in 20 mL of each solution and the theoretical amount of $CaCO_3$ that can be obtained by reacting 20 mL of each solution with excess sodium carbonate. Enter the results in the table.
5) Excess sodium carbonate solution (precipitant) is recommended to ensure that all of the calcium ions in solution are converted to product. For each sample, determine the volume of 0.5 M sodium carbonate solution that provides the stoichiometric number of moles of Na_2CO_3 needed to react completely with the $CaCl_2$ solution. Multiply the result by 1.2 to provide a 20% excess, and enter the results in the table.
6) Calculate the theoretical water hardness in mg $CaCO_3$/L for each water sample. Recall that each sample has been concentrated by a factor of 100 to provide the solution shown in column 2. The calculation for sample 1 is shown below as a guide.

   Sample Calculation (Sample 1): $\frac{0.801 \text{ g } CaCO_3}{0.020 \text{ L}} \text{ x } \frac{1000 \text{ mg}}{1 \text{ g}} \text{ x } \frac{1}{100} = 400 \text{ mg } CaCO_3/L$

   Note that the factor 1/100 accounts for the concentration of the original water sample to the final analyzed volume of 20 mL.

When you complete the rest of your Pre-Lab Assignment, be sure to sketch your procedure and include the reagents needed (reagents list), the glassware and equipment that will be used (materials list), and the appropriate measurements and observations that must be made (blank data tables).

<u>Data Analysis</u>:
Calculate the percent yield of calcium carbonate and determine the experimental water hardness in mg/L for each sample. Classify the water hardness of each sample according to the criteria established by the U.S. Department of the Interior and the Water Quality Association (see the Background section). Compile the class data for all the samples that were analyzed and copy the percent yield values for each sample (there may be more than one) into your lab notebook.

<u>Conclusion</u>:
Compare the accuracy and sensitivity of the gravimetric analysis procedure over the range of possible water harness from 50 to 500 mg/L. Identify three sources of error in this lab and explain how each error would quantitatively affect your results. Suggest two ways to reduce these sources of error.

<u>Post-Lab Questions</u>: Answer these after your Conclusion in your lab notebook.

Copper(II) chloride ($CuCl_2$; 0.98 g) was dissolved in water and a piece of aluminum wire (Al; 0.56 g) was placed in the solution. The blue color due to copper(II) chloride soon faded and a red precipitate of solid copper was observed. After the blue color had disappeared completely, the leftover aluminum wire was removed from the solution and weighed. The mass of the leftover aluminum wire was 0.43 g.

1) Calculate the number of moles of (a) copper(II) chloride and (b) aluminum that reacted.
2) What is the mole ration of copper(II) chloride to aluminum metal? Express this to the nearest whole number ratio.
3) What happened to the aluminum metal that was consumed in this reaction? Write the formula of the most probably aluminum-containing product.
4) Write a balanced chemical equation for the single replacement reaction of copper(II) chloride with aluminum.