3. HOW HARD IS YOUR TAP WATER?

Introduction

The quality of tap water is an important consideration in all communities. Hard water leaves inorganic build-up in pipes and water supply systems that is commonly referred to as *limescale*. Water becomes hard when ions are dissolved in it and the extent of hardness varies from community to community across the country. This lab uses gravimetric analysis to quantitatively determine the hardness of samples of tap water. A second analytical procedure will be performed using conductivity and a comparison of data obtained from both methods will be made.

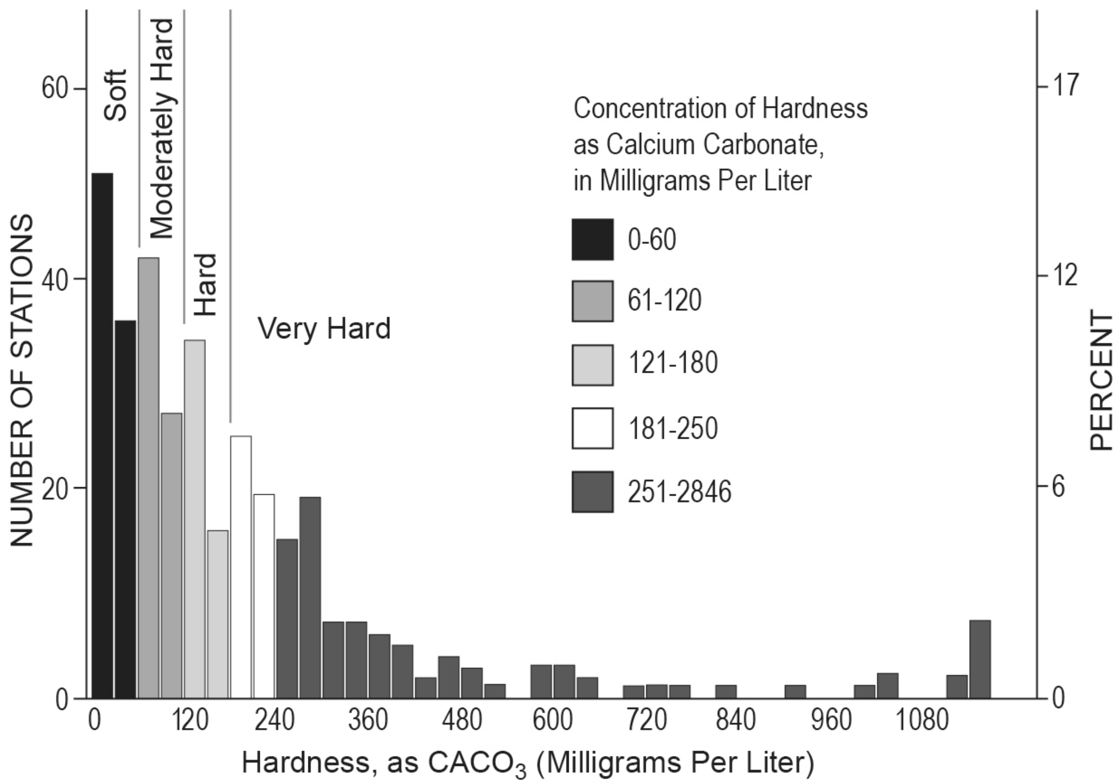
Concepts

- Gravimetric analysis
- Double replacement reactions
- Precipitate
- Water hardness
- Stoichiometry
- Conductivity

Background

Have you ever noticed a white, scaly build-up around your faucets? Is there a cream-colored ring forming in your bathtub? These are the result of having hard tap water. Depending on where you live, natural water sources will contain different amounts of dissolved minerals. Water becomes hard when these ionic compounds dissolve and separate into their component cations and anions in solution. The most common cations found in hard water are $Ca^{2+}$and Mg^{2+} and to a lesser extent, Fe^{3+} while chloride, sulfate, phosphate and carbonate are the most common anions. Hard water is one of the most common water quality problems homeowners encounter. It reduces the life of appliances, leaves filmy soap scum across bathrooms and kitchens, and dries out hair and skin. With more than 85% of households in the United States relying on hard tap water for their cooking, cleaning and bathing, many have turned to water-softening systems to help improve water quality. Water-softening systems remove calcium and magnesium ions using ion-exchange resins charged with sodium ions. In essence, ion-exchange replaces ions that cause hardness with ions that do not. U.S. governmental agencies classify water hardness in units of milligrams $CaCO_3$ per liter water (mg/L) or parts per million (ppm).

Classification	mg/L or ppm	grains/gallon
Soft	0-17.1	0-1
Slightly had	17.1-60	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



In this lab, you will be investigating the hardness of tap water using gravimetric analysis and conductivity. Gravimetric analysis is a quantitative analytical method that measures the amount of a specific analyte (an ion being analyzed) based on its mass. It remains one of the oldest and most widely used quantitative methods to determine the amount and identity of an unknown substance. The process involves transforming the analyte into a water insoluble form which precipitates out of solution and can be isolated by filtration and drying. The mass of the precipitate and stoichiometric considerations allows one to calculate the quantity of the analyte in the original sample. Conductometric titration is a type of titration in which the electrolytic conductivity of a reaction mixture is continuously monitored as one reactant is added. The equivalence point is the volume of added titrant at which conductivity undergoes a sudden change. A marked increase or decrease in conductance is associated with the changing concentrations of the most highly conducting ions (e.g. hydrogen and hydroxyl ions in the case of an acid-base reaction) and is useful in monitoring reactions that involve colored species that would otherwise be difficult to study using classical color indicators. In this lab, electrical conductivity will be compared to gravimetric methods in determining the calcium content of aqueous samples.

Pre-Lab Questions

1. Consider the reaction between aqueous solutions of lead(II) nitrate and sodium sulfate. Write the complete balanced equation for this reaction.

2. Write the complete and net ionic equations for this reaction.

3. If an antacid tablet containing calcium carbonate requires 75.85 mL of a 0.70 M HCl solution to neutralize it as shown in the equation below. Calculate the mass of calcium carbonate contained in the tablet.
   $CaCO_3(s) + 2HCl(aq) \rightarrow CaCl_2(aq) + CO_2(g) + H_2O(l)$

4. Given two solutions: 0.10 M $CaCl_2$ and 0.10 M Na_2CO_3, write the complete balanced equation for the precipitation reaction then calculate the volume of each solution needed to produce 1.50 g $CaCO_3$ precipitate.

Materials and Equipment

Use the following materials to complete the initial investigation. For conducting an experiment of your own design, check with your teacher to see what materials and equipment are available.

- Data collection system
- Wireless conductivity sensor
- Wireless drop counter with included accessories (syringe, drop tip, clamp, micro stir bar, stopcocks)
- Magnetic stirrer
- Precision balance (readability: 0.001 g)
- Beakers, 100-mL (3)
- Beakers, 250-mL (2)
- Graduated cylinders, 25-mL (2)
- Graduated cylinder, 50-mL
- Watch glass
- Stirring rod
- Funnel with clamp
- Ring stand
- Drying oven (class use), optional
- Filter paper
- 0.10 M sodium carbonate (Na_2CO_3) solution
- 0.10 M calcium chloride ($CaCl_2$) solution
- Unknown Solutions: A, B and C
- Various hard water samples, concentrated
- Distilled water in wash bottle
- Waste beaker

Safety

Follow these important safety precautions in addition to your regular classroom procedures:

- Wear safety goggles and gloves at all times.
- Solid calcium chloride may be hazardous when ingested and generates heat when solubilized in water.
- Sodium carbonate causes eye irritation and may be hazardous if ingested.
- Wash hands thoroughly with soap and water before leaving laboratory.
- Dispose of solutions as directed by Material Safety Data Sheet.

Disposal

If your drain system is connected to a sanitary sewer system, the following instructions apply. Calcium chloride, sodium carbonate and calcium carbonate may be rinsed down the drain with an excess of water. If your drain system does not empty into a wastewater facility, non-flammable and inorganic aqueous waste must be disposed of in a landfill or via a licensed hazardous waste provider.

Initial Investigation

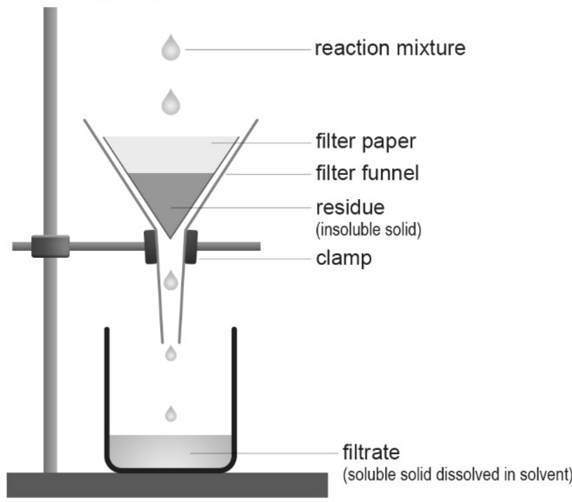
Precipitation Reaction and Gravimetric Analysis

1. Obtain the appropriate volumes of 0.10 M $CaCl_2$ and 0.10 M Na_2CO_3 from your instructor that should give you a theoretical yield of 0.500 g $CaCO_3$. Use calculations in Pre-Lab Question # 4 for guidance.

2. Mix the two solutions in a 250-mL beaker, stirring with a glass rod.

3. Measure the mass of a filter paper then fold it into fourths to form a cone. Fit the filter into a filtration apparatus as shown below. Use a 250-mL beaker to collect the filtrate.

FILTRATION



4. Pour reaction mixture through the filter apparatus to collect the precipitate. Rinse the reaction beaker with distilled water to collect as much of the precipitate as possible.

5. Record the mass of a watch glass.

6. Transfer the filter paper containing the precipitate to the watch glass and allow to dry for at least 24 hours. Alternatively, the watch glass may be placed in a drying oven set at 90 °C for 20-30 minutes.

7. Record the mass of the watch glass and filter paper after the drying process. Determine the mass of the precipitate product.

8. Dispose of the precipitate and filter paper as instructed by your teacher.

9. Calculate the percent yield and percent error of product recovery.

Conductometric Titration of Calcium Chloride with Sodium Carbonate

1. Open the *03 How Hard is Your Tap Water?* lab file. If the lab file is not available, create a graph of Conductivity versus Fluid Volume (mL) after the following step.

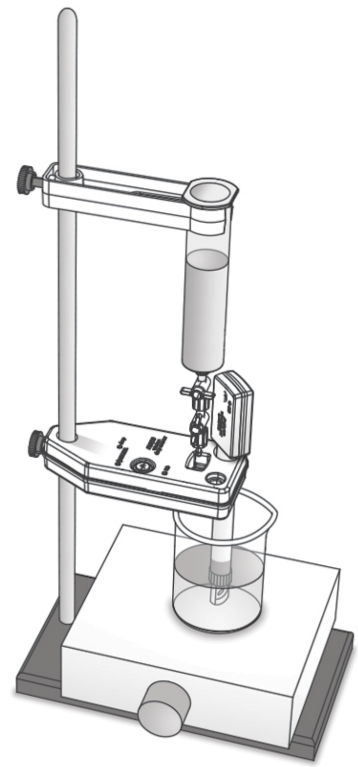
2. Connect the conductivity sensor and drop counter to your device.

3. Attach the drop counter to the ring stand.

4. Remove the plunger from the syringe. Attach both stopcocks and drop tip. Set both stopcocks to the closed (horizontal) position. To prime the syringe and eliminate air bubbles, open both valves (vertical position) and rinse with a small volume of 0.10 M Na_2CO_3 into a waste beaker.

5. Close both valves. Fill the syringe reservoir to the 50 mL mark with 0.10 M Na_2CO_3. Attach the syringe to the ring stand.

6. Set the waste beaker beneath the syringe. Fully open the bottom valve. Slowly open the top valve to achieve a drop rate of approximately 1 drop per second or slower. Once the desired drop rate is achieved, use only the bottom valve to open and close the syringe. Attach the syringe to the ring stand as shown.



7. Set a 25-mL graduated cylinder beneath the drop counter. Arrange the syringe to allow drops to fall through the drop counter directly into the graduated cylinder. Calibrate the drop counter with approximately 10.0 mL of titrant.

8. Set up the conductivity sensor and stir plate as shown. Secure the micro stir bar to the probe tip and make sure the magnet will not strike the probe while spinning.

9. Measure 15.0 mL of the 0.10 M $CaCl_2$ solution and transfer into a 100-mL beaker. Add enough distilled water to the beaker to fully immerse the tip of the conductivity probe as shown.

10. Turn on the stir plate and start collecting data.

11. Open the bottom syringe valve and monitor data collection. Conductivity will decrease gradually, then it will begin to increase. Once conductivity begins to increase, allow the titration to proceed for an additional 2-3 mL of titrant, then stop collecting data.

12. Stop the titration and dispose of reaction materials as directed by your teacher. Rinse the probe and micro stir bar.

13. From your titration data, determine the equivalence point of the reaction and calculate the molarity of the $CaCl_2$ solution. From this concentration, determine the mass of calcium carbonate precipitate present in the beaker and calculate the percent error from theoretical values. Based on your results, which method is best for water Ca^{2+} analysis: gravimetric analysis, or conductometric titration?

Advanced Investigation

Calcium Ion Concentration in Aqueous Solutions using Conductometric Titration

1. Obtain two Unknown water samples from your instructor. Record each sample letter (A, B, or C).

2. Refill the syringe reservoir to 50 mL with 0.1 M Na_2CO_3. If necessary, refer to the setup instructions in the previous section to achieve the desired drop rate. Calibrate if necessary.

3. Secure the micro stir bar to the conductivity probe tip and make sure the magnet will not strike the probe while spinning.

4. Measure 15.0 mL of the Unknown solution and transfer into a 100-mL beaker. Add enough distilled water to immerse the tip of the conductivity probe.

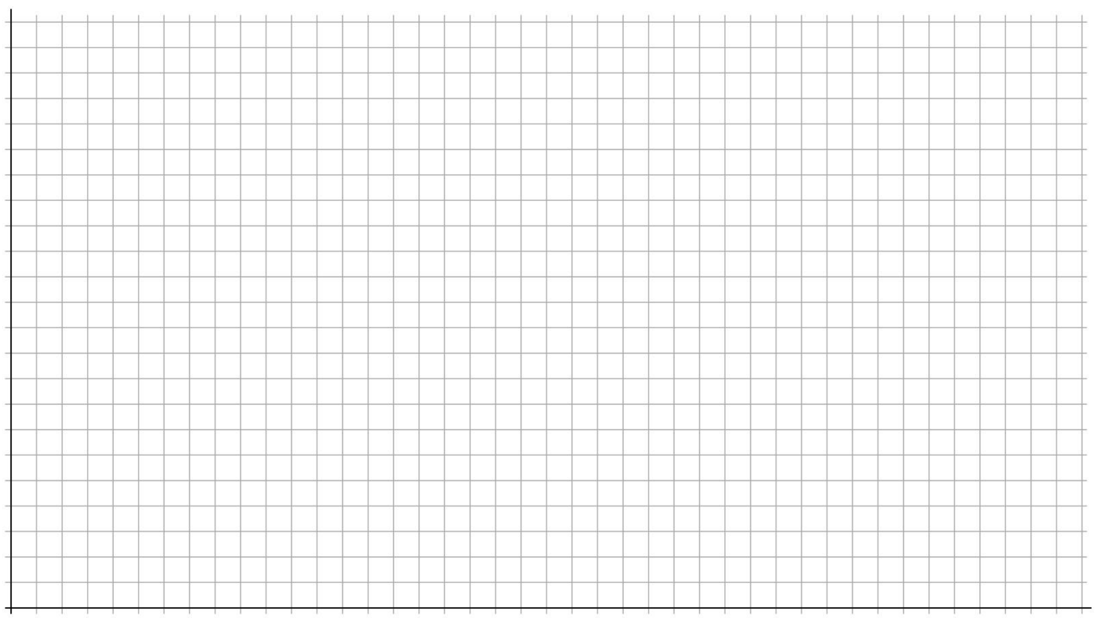
5. Turn on the stir plate. Start data collection and open the bottom valve.

6. Conductivity will decrease gradually. Monitor data collection until conductivity begins to increase. Allow the titration to proceed for an additional 2-3 mL of titrant, then stop collecting data.

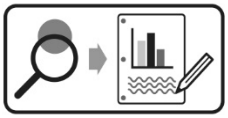
7. Stop the titration and dispose of reaction materials as directed by your teacher. Rinse the probe and micro stir bar.

8. Perform a titration of the second Unknown water sample. Dispose of all materials as instructed by your instructor.

9. For each Unknown sample, determine the equivalence point of the reaction, calculate the molarity of $CaCl_2$, and determine percent error from theoretical values provided by your teacher.



Extended Inquiry Investigation



Conductometric Titration of Tap Water Samples

Design a procedure to investigate the hardness (calcium and magnesium content) of tap water from different geographic areas. The water will have been concentrated by a factor of 20 by your instructor. Using the protocols detailed in the Initial Investigation, select either gravimetric analysis or conductimetric titration for this analysis. Discuss the procedure with your instructor before proceeding.

Gravimetric Analysis of the Calcium Content of an Antacid Tablet

The calcium content of a drugstore antacid tablet may also be determined by gravimetric analysis. These tablets contain many ingredients that act as binding agents and are inert. Develop a procedure to isolate the calcium carbonate from binding agents and determine the amount of calcium.

Synthesis Questions

1. Which other cation, if any, could be used instead of Ba^{2+} to form a precipitate with SO_4^{2-} ions?

2. How would you determine the percent sulfate content in a $CaSO_4$ sample, which is not very water soluble?

AP® Chemistry Review Questions

In an experiment, you are tasked with determining the mass of SO_4^{2-} in an alkali sulfate sample by precipitating with Ba^{2+} ions to form barium sulfate ($BaSO_4$). By obtaining the mass of the dried precipitate, you can calculate the SO_4^{2-} content of the sample.

A 0.355 g sample of an alkali sulfate was dissolved in 50 mL of water in a 400-mL beaker. Five mL of 6 M HCl was added, followed by an additional 200 mL of water. The solution was heated to 90 °C then 25 mL of $BaCl_2$ was added.

The precipitate was filtered then dried thoroughly and its mass was determined to be 0.611 g.

1. Assuming that all of the precipitate is $BaSO_4$, what is the mass of SO_4^{2-} in the precipitate?

2. Calculate the mass percent SO_4^{2-} of the unknown alkali sulfate.

3. What are the molecular, complete ionic and net ionic equations of the reaction of the sample with $BaCl_2$? Use M^+ to symbolize the metal ion of your unknown alkali sulfate.