VALENCIACOLLEGE

Experiment 7
Analysis of a Gaseous Product

Version 5

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A mixture containing calcium carbonate is reacted with hydrochloric acid. The carbon dioxide gas produced is captured and measured. A gas law and stoichiometric relationships allow for the determination of the percent of calcium carbonate in the mixture analyzed.

Objectives

- Determine the percent by mass of calcium carbonate in the garden lime under investigation.
- Measure pressure and become familiar with units of pressure.

Learning Outcomes

- Understand the nature of units of measurement and apply proper rules of significant figures to measurements and calculations.
- Understand and apply the relationships of physical behavior of gases.
- Enhance student's understanding of the application of the scientific method to solve complex problems.
- Understand and apply stoichiometric relationships.
- Understand and apply the relationships of physical behavior of gases.
- Perform essential lab techniques in laboratory setting.

Definitions

- **Alkaline** – a term referring to pH values above 7 when the solution is basic (related to the amount of hydroxide, OH^-, in the solution)
- **Analytical chemistry** – a field of chemistry that involves methods or instruments for separating, identifying, and determining amounts of compounds in a sample
- **Dalton's Law of Partial Pressure** – the total pressure of a gas mixture (P_t) equals the sum of the individual gas pressures ($P_t = P_A + P_B + P_C + \ldots$)
- **Environmental chemistry** – area of chemistry that focuses on chemical processes that occur in the environment and their impact on the environment
- **pH** – a scale that specifies the acidity of a solution; values typically run from 0-14, above 7 being basic, and below 7 being acidic
- **Relative percent error** – the absolute value of a calculation that indicates the accuracy of an experimentally determined value against the true, correct, or accepted value
- **Standard deviation** – a calculation that indicates the precision of a series of values by showing the variation around the average or mean
- **Volatile** – easily evaporates at normal temperatures

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Introduction

The lawns of City Hall were destroyed. All the grass died in a matter of days. Upon investigation, the groundskeeper found that the lawn had been recently treated with Green-Thumb, Inc.'s garden lime. The pH of the soil was tested and found to be too high. For the last three years, City Hall has been using Green-Thumb, Inc.'s fertilizers, weed-killers, plant nutrients, pH soil kits, and garden lime. They have never had problems before this one! The Beautification Manager requested Green-Thumb, Inc. pay for the damages, but they refused. The company stands by their product and says that the formulation was correct. The Beautification Manager contracted lawyer Bergeron to sue the company for damages. The lawyer has subcontracted Dr. Nakamura, an environmental researcher from Valencia College, to analyze the garden lime in question. The groundskeeper has provided the lawyer with several bags of the garden lime. The label on the garden lime states that it is 70% calcium carbonate. Acceptable industry tolerance for this type of product ranges within $\pm$ 5% of the value stated on the label.

You are a graduate student pursuing your Ph.D. in analytical chemistry, doing research in Dr. Nakamura's lab. Since you have a master's degree in environmental chemistry, she has assigned your research team to determine the percent of calcium carbonate in the garden lime in question. As an environmental chemist, you know that the scale that measures pH runs from 0 to 14; neutral soil has a pH of 7. However, many factors can change pH from neutral including irrigation water, vegetation, and the parent material from which the soil is made. Above 7, soil is increasingly alkaline; below 7, it is increasingly acidic. Most plants grow best in slightly acidic soil with a pH of 6.5.[1] Garden lime is added to soils to reduce acidity; solid ground sulfur, iron(II) sulfate, or aluminum sulfate are added to reduce alkalinity.[1]

Garden lime is made from pulverized limestone[1]. Calcium carbonate (also called calcite) is the principal mineral found in limestone[1]. In addition to limestone, it is the principal mineral found in marble, chalk, pearls, stalactites, stalagmites, caliche, and the shells of marine animals such as clams.[2] It is also the principle mineral responsible for hard water. Calcium carbonate is an alkaline compound so it readily reacts with acidic media. Carbon dioxide is produced by this reaction:

$$CaCO_3(s) + 2\,HCl(aq) \rightarrow CaCl_2(aq) + H_2O(l) + CO_2(g) \qquad \textit{Equation 1}$$

The goal of this experiment is to determine the percent by mass of calcium carbonate in the garden lime under investigation. To accomplish this, you will treat a small sample of the garden lime mixture with an excess of 3 M hydrochloric acid, and collect the carbon dioxide gas produced. With proper measurements and stoichiometric relations, you will determine the percent of $CaCO_3$ in the sample.

- Determining the **volume** of CO_2 collected: If the CO_2 is collected in a flask containing water, the CO_2 will displace water equal to its volume (See Figure 1).

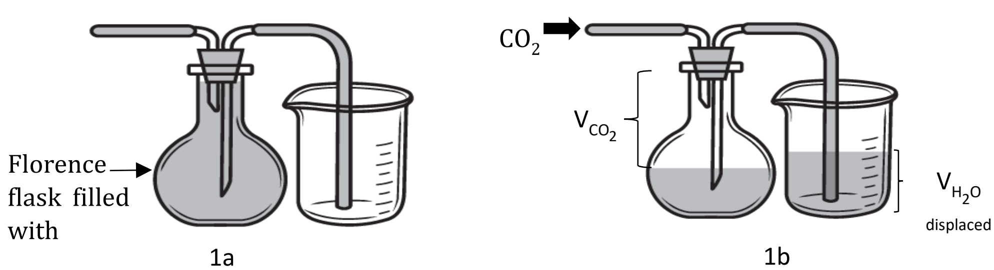

Figure 1. Florence flask used for gas collection.

- Determining the **pressure** of the CO_2 collected:
  Since water is volatile, the gas collected over water will be a mixture of two gases: water vapor and CO_2. Using Dalton's Law of Partial Pressure, the total gas pressure collected in this experiment will be:

$$P_t = P_{CO_2} + P_{H_2O} \qquad \textit{Equation 2}$$

The atmospheric pressure of the room (P_{atm}) is equal to the total pressure in the flask, P_t, if the water level below the gas is physically held at the same height as that of the water displaced (see Figure 3). To understand the reason behind this, consider the U-tube illustrated in Figure 2. A U - tube is a hollow glass tube in the shape of the letter U. If the tube is empty and we pour mercury into the right arm (Figure 2a), when we stop pouring the mercury, the level of the mercury in the left arm will be equal to the level of the mercury in the right arm (Figure 2b). Why? Because both arms are open to the environment, therefore the pressure on both arms is equal.

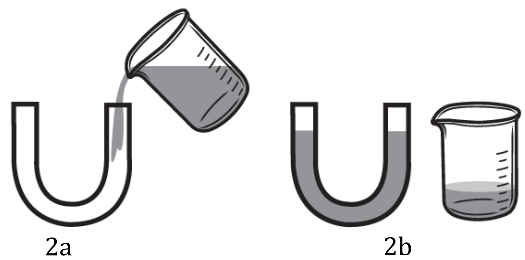

Figure 2. Mercury poured into a u-tube.

Now consider Figure 3a. Since the height of the volume in the beaker is higher than the height of the volume in the Florence flask, we cannot assume that the external pressure (atmospheric pressure) is the same as the gas pressure in the flask. When the flask is lifted, as shown in Figure *3*b, the pressures are equalized. This causes a small change in the volume of the water displaced (that is why it is important to keep the hose below the water level in the beaker); volume and pressure are inversely proportional. This shift in liquid, though not detectable visibly since both the flask and beaker are wide, is desirable, because you want to know the amount of water displaced when P_t is equal to the atmospheric pressure. (Think of an old movie when a criminal places a hose into a car's gas tank valve and suctions: when the hose is lowered the gasoline gushes out, but when the hose is lifted the gasoline stops flowing.)

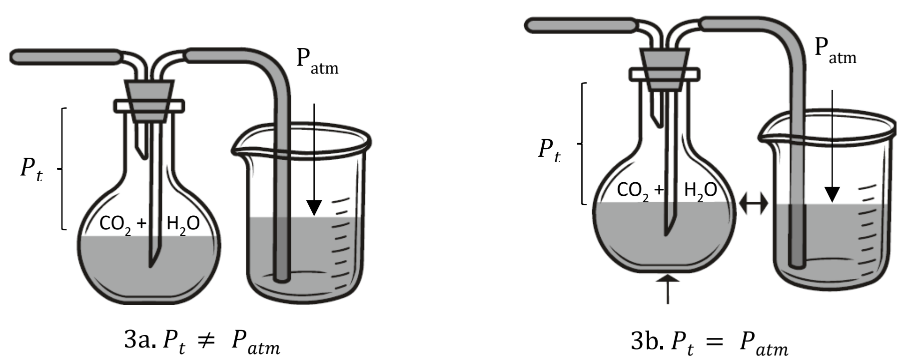

Figure 3. Adjusting the pressure in the flask by raising the Florence flask.
(If the height of the volume in the beaker is lower, raise the beaker.)

The atmospheric pressure is measured using a barometer (it is probably located on the professor's demonstration bench). The vapor pressure of water is dependent on the temperature (see Appendix 5 Vapor Pressure of Water at Various Temperatures).

Following are some useful conversion factors involving pressure:

1 atm = 760 mm Hg = 760 torr = 29.92 inch Hg = 101.3 kPa

- Determining the **temperature** of CO_2 collected:
  Since the CO_2 is in contact with the water in the Florence flask, its temperature is the same as the temperature of the water displaced.

- Determining the **percent by mass of $CaCO_3$** in the garden lime sample:
  Notice the relationship between CO_2 and $CaCO_3$ (Equation 1). Now consider the gas laws in the Gas Chapter of your class textbook (Boyles' Law, Charles' Law, Avogadro's Law, Ideal Gas Law, and Guy-Lussac's Law). Which one should you use to calculate the moles of carbon dioxide produced in this experiment? How will you determine the mass of $CaCO_3$ that reacted?

  The percent by mass of calcium carbonate can be calculated using Equation 3.

$$\text{Percent } CaCO_3 = \frac{mass\ CaCO_3,\ \ g}{mass\ garden\ lime,\ \ g} \times 100 \qquad \textit{Equation 3}$$

You will perform the analysis in triplicate, and report the mean percent by mass of calcium carbonate and the standard deviation of the percent by mass of calcium carbonate (see Equation 4 and Equation 5 below and Appendix 8.).

$$\text{Mean:} \qquad \bar{x} = \frac{x_1+x_2+x_3}{n} \qquad \textit{Equation 4}$$

$$\text{Standard deviation: } s = \sqrt{\frac{\sum(x_i-\bar{x})^2}{n-1}} \qquad \textit{Equation 5}$$

where: n is the number of trials
x_i is the individual result of each trial

In addition, you will calculate the relative percent error using Equation 6 (see Appendix 8), and state if the sample is within acceptable results or not.

$$Relative\ percent\ error = \left|\frac{true\ value - calculated\ value}{true\ value}\right| \times 100 \qquad \textit{Equation 6}$$

Example 1: Determining the percent of calcium carbonate in garden lime sample 37:

Sample 37 was analyzed in triplicate following the setup shown in Figure 4. The mass of the garden lime for Trial 1 was 2.195 g. After mixing it with HCl(*aq*), 325 mL of water were displaced at 23.6 °C. The atmospheric pressure (displayed by the barometer in the room) was 29.86 inches Hg. The vapor pressure of water was found by using Appendix 5. The sample was analyzed two more times. Following are the results for the percent by mass of calcium carbonate calculated for each trial:

Trial 1	Trial 2	Trial 3
59.0 %	57.8%	61.4%

The mean, the standard deviation, and the relative percent error were calculated.

Conclusion: The percent of $CaCO_3$ in sample 37 is 59.4% $\pm$ 1.8%. The relative percent error is 15.1%. The mean % $CaCO_3$ in Sample 37 is not acceptable.

(You are asked to verify these results in Prelab Question 3.)

Techniques

Technique

- Technique 1: Cleaning glassware
- Technique 2: Using a balance
- Technique 3: Transferring liquids
- Technique 4: Using a graduated cylinder
- Technique 11: Disposing chemical waste
- Technique 18: Measuring temperature

List of Chemicals

- Alka-Seltzer
- garden lime
- 3 M hydrochloric acid

List of Equipment and Glassware

- one 25-mL buret
- one 50-mL beaker
- one 250-mL beaker labeled as waste
- one 1000-mL Pyrex beaker
- one 2000-mL plastic beaker (or equivalent)
- one Florence flask
- one two-way connective tubing stopper
- one thermometer
- one 1000-mL graduated cylinder
- one small funnel
- one glass rod
- one small side-arm flask
- one watch glass

Experimental Procedure

Part A Sample Preparation and Apparatus Setup

1. **Water Saturated with CO_2.** Add about 1500 mL of tap water into a 2000-mL plastic beaker. Saturate this water with carbon dioxide by adding ¼ of an Alka-Seltzer tablet. Stir with a glass rod. Once the Alka-Seltzer has dissolved, try to remove as many gas bubbles as possible by stirring the water with the glass rod or tapping it gently.

Technique 2

2. **Sample preparation**.
    a. Obtain sample of the garden lime. These are located on the professor's bench (or other designated area).
    b. Record the sample number on the Experimental Data and Calculations sheet. Measure approximately 2.3 g of the sample. Record the mass in Data Table 1.
    c. Transfer the sample into a small side-arm flask. Set this flask aside for the moment.
3. **Set-up of CO_2 collection system.**
   Set up the CO_2 generator and gas collection apparatus as shown in Figure 4:
    a. Completely fill the Florence flask with the CO_2-saturated water prepared in step A1.
    b. Place the stopper with the connective tubing ensemble in the Florence flask (you might want to do this over the sink or place paper towels under it since it will overflow). Careful that you do not move the flask vertically. If you do, you will produce a siphon (water will flow out of the tubes).
    c. Make sure there is no air trapped under the stopper. (It is OK if the air bubbles are small, otherwise remove the stopper, add more water, and place stopper on again.)
    d. Notice the difference in length of the two glass tubes **below** the stopper on the connective tubing ensemble. Connect the hose coming from the glass with shortest length (below the stopper) to the side-arm flask (first secure the flask to the stand with a utility clamp).
    e. Place a pinch clamp through the hose coming from the glass tube with the longest length, then place this hose in a 1000-mL Pyrex beaker. Make sure the tip of the hose rests on the bottom of the beaker and the clamp rests in such a way that it does not pull the hose out of the beaker.

Technique 3

4. **Acid delivery system**.
    a. Obtain a 25-mL buret with a stopper. Place the buret on top of the side-arm flask to make sure the stopper fits tightly in place. If it doesn't, select another buret with stopper until you find one that fits well.
    b. Remove buret from flask.
    c. Pour about 30 mL of 3 M HCl in a 50-mL beaker.
    d. Rinse the buret (use a small funnel) with three small portions of 3 M HCl, making certain that the solution wets the entire inner surface. Drain each rinse through the buret tip.
    e. Fill the buret with HCl using a small funnel. (The purpose of the buret is to deliver the HCl in a closed system; the HCl will be the excess reagent, so you do not need to monitor the volume added.)
    f. Remove the funnel (place on a watch glass or in a beaker; do not place it directly on the bench). Secure the buret on stand. Place it on the side-arm flask when you are

Golden Lab Rules:

1) Label the HCl beaker.
2) Collect the rinses in the waste beaker.
3) Cover the remaining HCl with a watch glass.

ready to start the reaction. (You might find it easier to remove the utility clamp at this point.)

g. Make sure all connections are tight. The gas generated should not escape the system.

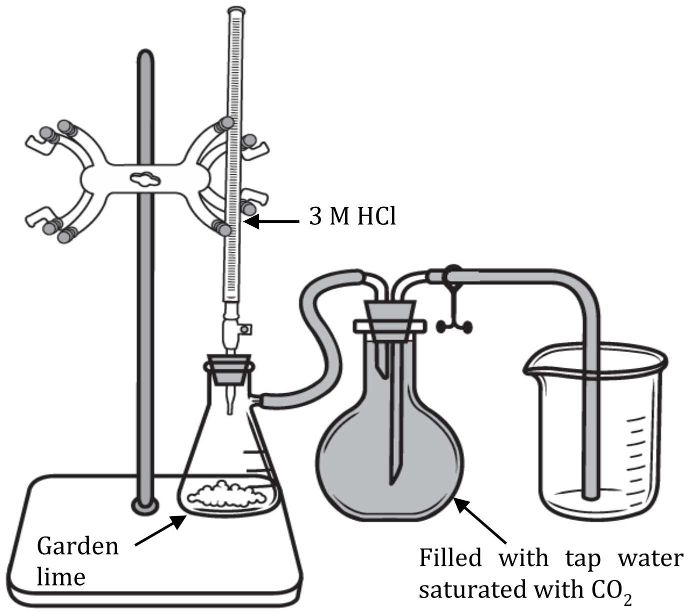

Figure 4: CO_2 Generator and Gas Collection Apparatus.

(Not to scale.)

Part B Collection of the Carbon Dioxide

1. Make sure that the **pinch clamp is open** and the hose in the 1000-mL beaker rests on the bottom of the beaker throughout the reaction.
2. Add small portions of HCl to the side-arm flask by turning the valve on the buret a complete full circle. Do not leave it open because the gas will escape through the buret. Add at least 6 mL of HCl.
3. Continue to add HCl as needed, but do not add so much that the HCl reaches the arm of the side-arm flask – you do not want the acid to go into the tubing.
4. Gently shake the side-arm flask to ensure that the acid continues to react with the calcium carbonate in the garden lime.
5. The reaction is complete when, upon addition of HCl and stirring, no more gas bubbles are produced.

Part C Measurement of the volume, temperature, and pressure of the CO_2

1. **Equilibration of the internal pressure** in the Florence flask with the room pressure:
   a. At this point, you have two containers with water: the 1000-mL beaker and the Florence flask. Which volume is lower, the one in the flask or the beaker? Physically raise the one that has the lower volume until the top layer of the water is as high as the top layer of the water in the other container. See Figure *3*.

b. As soon as the levels are at the same height, open the pinch clamp, and place the hose between the clamp legs so that it closes (pinches) the hose. Place the container back on the tabletop.
c. Remove the hose from the beaker.
d. Write your signature in Data Table 2 to indicate that you completed this adjustment.

2. **Temperature**: Measure the temperature of the displaced water and record it in Data Table 2.
3. **Volume**: Pour the water displaced into a 1000-mL graduated cylinder. Read and record the volume in Data Table 2.

Technique 18
Technique 4

4. **Barometric Pressure**: Determine the room pressure using the barometer located on the professor's bench. Record the value in Data Table 3. This pressure is equivalent to the pressure of the CO_2 plus water vapor in the Florence flask because the pressures were equilibrated prior to removing the hose.
5. Remove the side-arm filtration flask and pour the contents into the Acid/Base Waste container. Rinse it well with deionized water. Refill the Florence flask with the CO_2-saturated water (you may reuse the water that is in it as well as the water that was displaced).
6. Determine if you need to refill the buret with HCl. The next Trial should take approximately the same amount as the first Trial. If there is not enough HCl, add some more.
7. Repeat above procedure for Trials 2 and 3.

Part D Calculations

1. Record the vapor pressure of water in Data Table 3 and convert to the units requested in the Table. Calculate the pressure of the CO_2 collected for each trial. Show calculations for Trial 1 under the Table.
2. Design and complete Table 4, keeping in mind that your goal is to determine the percent of $CaCO_3$ for each trial. You might not need all the rows in Table 4. Show calculations for Trial 1 in the space provided.
3. Calculate the mean % $CaCO_3$ and its standard deviation. Record in Data Table 5.
4. Calculate the relative % error of the mean % $CaCO_3$ with reference to the expected value. Record in Data Table 5. Show calculations in the space provided.

Clean up/Disposal

- When finished, pour the contents of the side-arm flask, buret, and any acid left, into your Waste beaker. Rinse them with water; collect the rinse in the Waste beaker.
- Transfer the contents of your Waster beaker into “Acid/Base Waste” container.
- Rinse the buret with water and allow it to drain through the tip. Place it, tip up, with the valve open, in the buret canister.

Technique 11

- Wash the side-arm flask with soap and water. Rinse with deionized water.
- Pour the CO_2-saturated water down the drain and dry the outside of the glassware. (Not necessary to wash -only exposed to CO_2-saturated water). Return all equipment and glassware to its original location.

Pre-lab

1. Read the Introduction and Experimental Procedure. List all the data that needs to be measured.
2. You will need to weigh approximately 2.3 g of the garden lime. Write a detailed procedure on how to weigh it.
3. Using the data in Example 1, perform the following calculations, showing all your work.
    a. Calculate the moles of carbon dioxide produced in Trial 1. *Hint:* Consider which gas law you will use for this calculation. Which measurements require unit conversion for the law? (See pages 2 - 4.)
    b. Calculate the mass of calcium carbonate in Trial 1.
    c. Verify that the % calcium carbonate in Trial 1 was calculated correctly.
    d. Verify that the mean % calcium carbonate in sample 37 was calculated correctly.
    e. Verify that the standard deviation was correctly reported.
    f. Verify that the relative percent error was correctly reported.
4. A pure sample of calcium carbonate produced 0.998 g CO_2. What was the mass of the calcium carbonate used?
5. How many mL of 3.0 M HCl are needed to completely react with 2.500 g of pure calcium carbonate? [Note: You will use less than this calculated volume of HCl for each trial. Think about the maximum volume of HCl you will need. To prevent waste, do not take more than what you need to perform the experiment.]

Post-lab

In the data/calculations section:

1. Include the data tables 1-5.
2. Complete calculations for data tables 1-5.
3. In data table 5, state if the garden lime that you analyzed has an acceptable percentage $CaCO_3$.

References

1. Klein, C. *Vegetable Gardening*. I-5 Publishing, LLC. UK. **2016,** 21, 30.
2. Beran, J. A. *Laboratory Manual for Principles of General Chemistry*, 6th ed. John Wiley & Sons: New York, NY, **2000**, 237-244.

VALENCIA COLLEGE

Experiment 7: Analysis of a Gaseous Product
Experimental Data and Calculations

Name: ______________________________________ **Date:** ___________________

Lab Partner: ______________________________________ **Section:** ___________

Garden Lime Sample Number ___________

Table 1. Mass of Garden Lime Sample

	Trial 1	Trial 2	Trial 3
1) Mass of weighing boat ( )			
2) Mass of weighing boat + sample ( )			
3) Mass of sample ( )			

Table 2: Determination of Temperature and Volume of the Carbon Dioxide Gas Collected

	*Trial 1	Trial 2	Trial 3
1) Adjusted water level after reaction done (student's signature)			
2) Temperature of CO_2 ( )			
3) Volume of CO_2 ( )			
4) Temperature of CO_2 (K)			
5) Volume of CO_2 (L)			

* Show calculations for Trial 1 here:

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Name: ____

Table 3: Determination of the Pressure of the Carbon Dioxide Gas Generated

	*Trial 1	Trial 2	Trial 3
1) Barometric Pressure (inches of Hg)			
2) Vapor Pressure of H_2O ( ) (Appendix 5)			
3) Barometric Pressure (atm)			
4) Vapor Pressure of H_2O (atm)			
5) Vapor Pressure of CO_2 (atm)			

* Show calculations for Trial 1 here:

Table 4. Percent by Mass of Calcium Carbonate in Garden Lime Sample # ____________

	*Trial 1	Trial 2	Trial 3

* Show calculations for Trial 1 on the next page.

Table 5. Results Summary (* Show calculations on the next page.)

1) Mean percent by mass of calcium carbonate	
2) Standard deviation of the percent by mass of calcium carbonate	
3) Relative percent error of the mean percent by mass of calcium carbonate	
4) Does this garden lime sample have an acceptable percentage $CaCO_3$? (Yes or no.)	

Name: ______________________________

* Show calculations for Table 4 Trial 1 and calculations for Table 5(mean, standard deviation, and relative percent error) here: