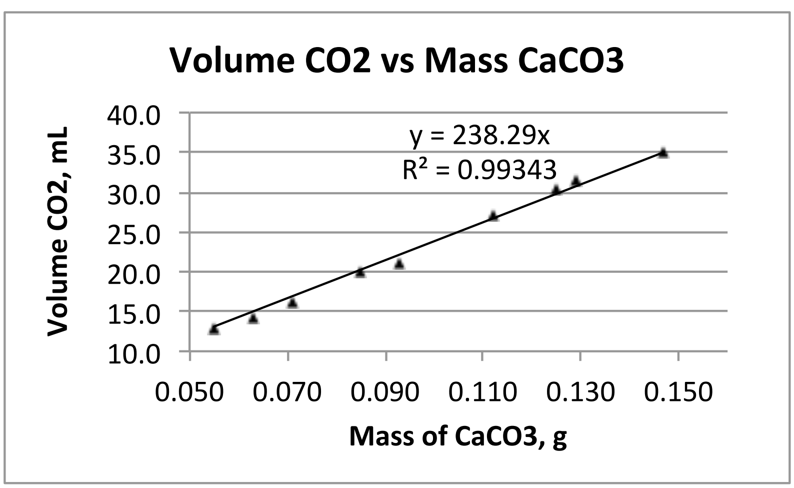
CHAPTER 7 PERCENT COMPOSITION

THIS CLASSROOM GROUP LABORATORY EXPERIMENT utilizes everyone's data to give an overall group result that demonstrates the concept of percent composition. A group graph is produced. This graph is used by each pair of students in Part 2 in order to determine the percent composition of a Tums® tablet. A second, more traditional method is also used so that the results can be compared.

In this chapter we describe a unique method of analysis that utilizes the volume of carbon dioxide generated by the acid decomposition of calcium carbonate samples inside the syringe. (The most common method for the analysis of a Tums tablet involves reacting the antacid with excess acid and back-titrating with base.) In Part 1 of this experiment students produce a graph as a group effort that shows the relationship between the mass of pure calcium carbonate used and the volume of carbon dioxide produced. The reaction is:

$$CaCO_3(s) + 2\,HCl(aq) \rightarrow CaCl_2(aq) + CO_2(g) + H_2O(l)$$



Volume of carbon dioxide produced as a function of mass of calcium carbonate used.

The group graph produced should look similar to graph at left. In Part 2 of the experiment, students react samples of Tums of known mass as they did with the pure calcium carbonate. The volume of carbon dioxide they obtain can be converted to mass of calcium carbonate with the use of the graph. Knowing the mass of the sample of Tums used and the mass of calcium carbonate from the graph, they can determine the percent calcium carbonate in Tums.

In Part 3, students perform the experiment using the general method of Slater and Rayner-Canham[1] in which the calcium carbonate content of an antacid is determined by three mass measurements: (a) the mass of the sample of crushed Tums; (b) the mass of the Tums sample plus the mass of a small quantity of 4 M HCl(aq) in a separate container; and (c) the mass of Tums + acid after they have been mixed and the reaction has taken place. The loss of mass is attributed to carbon dioxide. From there, the percent $CaCO_3$ in the Tums can be determined.

Analyses of Tums tablets by these two methods give similar results. In several trials, we have determined by the syringe method described (Part 2) that Tums are 36 – 39% $CaCO_3$. Using the traditional method (Part 3), we get 41 – 43% $CaCO_3$. We think two factors contribute to the discrepancy: (1) CO_2 is soluble in water so that the syringe method gives slightly low results and (2) The Tums tablets splatter water as they react with considerable fizzing; some water is undoubtedly lost this way in the traditional method, giving results that are slightly high. Nevertheless, the results are similar.

[1] ***Microscale Chemistry Laboratory Manual***, Alan Slater and Geoff Rayner-Canham, Addison-Wesley Publishers Ltd., 1994.

PERCENT COMPOSITION
INFORMATION FOR THE TEACHER

Suitability

For use by high school and university-level chemistry students. This experiment can be conducted at about the time that the concept of percent composition of mixtures is being introduced.

Background skills required

Students should be able to:

- generate a gas as learned in Chapter 1
- measure quantities of liquid reagents
- use a balance
- use a ruler
- accurately read the volume gradations on the syringe (including estimating between two marks)

Time required

Students should be able to perform this experiment in a single 45-minute laboratory period.

Equipment

Microscale Gas Chemistry Kit (Chapter 1)
top-loading balance
analytical balance
pipet (made into a spatula as described below)
4 L pail (plastic ice cream pail)

Chemicals

HCl (3.0 M)
$CaCO_3$(s)
Tums tablets

Before students arrive

Each experiment will consume 5 - 7 mL 3 M HCl(aq), but we suggest that the volume estimation be based on 8 – 10 mL per experiment. Prepare a bottle of approximately 3 M HCl(aq) by diluting 45 mL concentrated HCl with 135 mL water to give a total of 180 mL (or diluting 90 mL 6 M with 90 mL H_2O). Determine which pair of students will perform each experiment. Include the following approximate masses of pure calcium carbonate in the list of experiments to be done: 0.05 g, 0.06 g, 0.07 g, 0.08 g, 0.09 g, 0.10 g, 0.11 g, 0.12 g, 0.13 g, 0.14 g, and 0.15 g. Hint: Make an inexpensive "spill-proof" spatula for each pair of students by cutting the bulb off of a pipet at an angle:

An inexpensive spill-proof spatula

Vapor pressure of water

Adjusting for the vapor pressure of water is ***unnecessary*** because both Parts 1 and 2 are both performed in the same way and presumably at the same temperature. Technically, the graph produced in Part 1 should have the volume of CO_2 (y-axis) adjusted to subtract the volume attributed by water vapor. For example, at 20 ^oC, the vapor pressure of water is 17.5 mmHg, so if the external pressure were 740 mmHg at the time of the experiment, the adjusted volume of CO_2 would be (740 – 17.5)/740 = 0.976 of the value determined using the student instructions below. However, the volume of CO_2 determined in the Tums analysis would have to be similarly adjusted, and the net result would be the same as if no compensation for water vapor pressure were included.

Website

This chapter is available at our gas website:

http://mattson.creighton.edu/Microscale_Gas_Chemistry.html

Instructions for your students

For classroom use by teachers. Copies of all or part of this document may be made for your students without further permission. Please attribute credit to Professors Bruce Mattson and Mike Anderson of Creighton University and this website.

Content for this chapter first appeared as "Microscale Gas Chemistry, Part 24. Percent Composition of Calcium Carbonate in Tums®. A chemistry laboratory experiment.", Mattson, B.; and Saunders, E., ***Chem13 News***, **313**, September, 2003.

PERCENT COMPOSITION
INSTRUCTIONS FOR STUDENTS

General Safety Precautions

Always wear safety glasses. Gases in syringes may be under pressure and could spray liquid chemicals. Follow the instructions and only use the quantities suggested.

Toxicity

Carbon dioxide is relatively non-toxic; however, it is a simple asphyxiant if inhaled in very large quantities. We will not be generating large quantities of carbon dioxide.

Part 1. Class Calibration Curve

1. Using a *top-loading balance*, measure into a weighing dish the quantity of calcium carbonate that you and your lab partner were assigned to use.

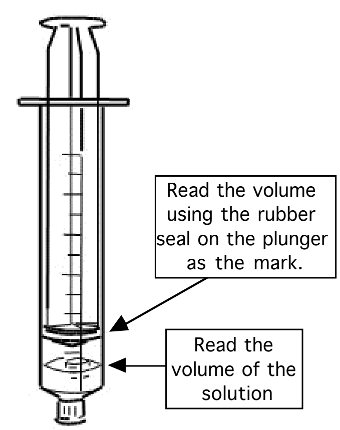
2. Place an empty vial cap on the *analytical balance* and tare the balance to read 0.0000 g. Remove the vial cap and carefully transfer the pre-measured $CaCO_3$(s) into the vial cap. Do not get any of the solid on the outside of the vial cap. Return the vial cap to the analytical balance and determine its exact mass. Record the exact mass on your Report Sheet.

3. Lower the cap containing the $CaCO_3$(s) into the syringe by flotation.

4. Measure out 6 – 8 mL 3 M HCl(aq) into a weighing dish.

5. Draw up 5 mL of the acid into the syringe. Push the syringe fitting into the syringe cap. Use caution so that the reagents do not mix until Step 7.

6. Read the initial volume of the syringe using the bottom of the rubber seal as the mark (see figure). Also read the level of the acid solution. The difference between these two readings is the volume of air in the syringe. This volume will be subtracted later. Record your data.



Reading the syringe.

7. Perform the reaction by shaking the syringe. The reaction is fast. Assist the plunger from time to time by pulling it outward by a few mL. The reaction is done within a few seconds, when no more bubbles are being produced in the solution.

8. You are now ready to measure the final volume. You'll get your hands wet doing it. First, pull the plunger outward until it feels like you are pulling against a force. Let go of the plunger and it will return to an "equilibrium" position where the pressure inside the syringe is fairly close to the outside pressure. Carbon dioxide tends to dissolve in water (as it does in carbonated beverages), so we must force it out of solution before we measure the volume of $CO_2(g)$. Here's how: Pull the plunger out by 10 mL or so from its equilibrium position and shake and tap the syringe vigorously. Notice that your action forces dissolved carbon dioxide to come out of solution with noticeable bubbling. Next, immediately remove the syringe cap underwater while holding the plunger outward creating a reduced pressure — use a large container such as an ice cream pail in order to accommodate your hands and the syringe. Remove the syringe cap deep enough under enough water so that only water — no air — enters the syringe. Water will rush into the syringe to equalize the pressure. Recap the syringe underwater. The gas pressure inside the syringe is now very close to the atmospheric pressure outside the syringe. Be careful to not move the plunger inward or outward after it has been recapped. Take the final volume readings for both gas and solution as previously done in Step 6. The difference in volumes this time is the volume of carbon dioxide + air initially present. The volume of carbon dioxide only is obtained by subtracting the volume of air (Step 6) from the volume of carbon dioxide + air just determined. Record all results. Note: We have included a QuickTime movie of this step at the website version of this article.[1]

9. You instructor will provide you with instructions for sharing the data with your classmates (such as plotting your results on a group graph).

Part 2. $CaCO_3$ in Tums tablet using class graph and volume of gas

Repeat the experiment (Steps 1 – 8) with a sample of Tums instead of pure calcium carbonate. Tums tablets consist of calcium carbonate and a number of other ingredients as listed on the bottle. Only calcium carbonate produces gas in the reaction with HCl(aq). Use a mass of approximately 0.25 – 0.32 g. The sample can be used as a chunk; it does not need to be pulverized. Record the exact mass used. Record the four volume readings as per Steps 6 and 8 above. You will notice that the Tums does not react quite as quickly and leaves a milky solution after the evolution of gas has ceased.

[1] Website: http://mattson.creighton.edu/Part24-Tums/Part24-Tums.html

Part 3. $CaCO_3$ in Tums tablet using the mass lost method

1. Place 15 mL 3 M HCl(aq) in a 250 mL plastic cup.
2. Determine the mass of a Tums tablet. (It will be approximately 1.3 – 1.4 g)
3. Place the cup of acid and the Tums tablet side by side on the balance and determine the total mass.
4. Remove the acid and Tums from the balance and add the Tums tablet to the acid. It will fizz as it releases CO_2(g). Wait until the bubbles have stopped forming. Swirling the cup will accelerate this process. Because CO_2(g) is heavier than air, tip the cup slightly to "pour out" the CO_2(g), but do not pour out any liquid.
5. Determine the mass of the resulting solution. The difference in mass between this and the mass determined in Step 3 is due to the CO_2(g) lost during the reaction. This can be converted into moles of CO_2(g). This also equals the moles of $CaCO_3$(s) — see equation above. One can then convert moles of $CaCO_3$(s) into mass of $CaCO_3$(s) and determine the % $CaCO_3$ in the Tums tablet.

Laboratory Report:

Part 1. Class calibration curve

Mass of pure $CaCO_3$(s) used: Volume of carbon dioxide calculation:

Initial syringe readings:

Rubber seal (mL): Solution (mL): Volume air (mL):

Final syringe readings:

Rubber seal (mL): Solution (mL): Volume air + CO_2 (mL):

Volume of CO_2 collected (mL):

Part 2. $CaCO_3$ in Tums tablet using class graph and volume of gas.

Mass of Tums used:

Volume of carbon dioxide calculation:

Initial syringe readings:

Rubber seal (mL): Solution (mL): Volume air (mL):

Final syringe readings:

Rubber seal (mL): Solution (mL): Volume air + CO_2 (mL):

Volume of CO_2 collected (mL):

Part 3. $CaCO_3$ in Tums tablet using mass lost.

Mass of Tums used:

Mass of cup of acid + Tums tablet before reaction (Step 3):

Mass of cup of acid + Tums tablet after reaction (Step 5):

Mass of CO_2 gas produced:

Disposal of carbon dioxide samples

Unwanted carbon dioxide samples can be safely discharged into the room.

Clean-up and storage

At the end of the experiments, clean all syringe parts (including the diaphragm), caps and tubing with soap and water. Rinse all parts with water. Be careful with the small parts because they can easily be lost down the drain. Store plunger out of barrel.

Questions

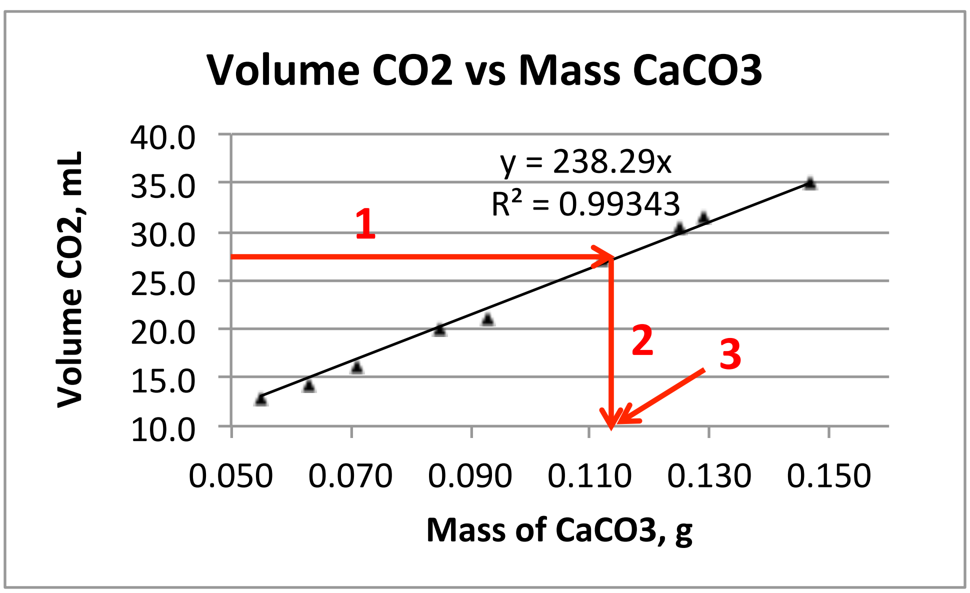
Part 1. Class calibration curve

1. Add your data points to the graph being prepared on the chalkboard (or follow the data collection procedures given by your teacher). Do your data agree with the general trend?

Part 2. $CaCO_3$ in Tums tablet using class graph and volume of gas

1. What mass of $CaCO_3$(s) was present in your Tums sample? After all of the data from all groups are available, you can now answer this question as described below. Your teacher will provide you with either (a) or (b):

   (a) A sketched line on the class graph for direct use. In this case, you should locate on the graph's y-axis the volume of CO_2(g) produced in your Tums experiment. (Step 1) Draw a horizontal line that intercepts the sketched line and (Step 2) drop a vertical line to the x-axis. (Step 3) This is the mass of $CaCO_3$(s) in your Tums sample. In the example below, the mass of $CaCO_3$(s) turns out to be 0.115 g.



(b) The equation for the line in the form of $y = mx + b$. The slope, $m = \Delta y/\Delta x$ where y = volume of CO_2 and x = mass of $CaCO_3$; can be estimated or calculate using a spreadsheet such as Excel. The y-intercept, b, should be 0. If your teacher provides you with the equation, it will look as follows, using the example shown in figure, page 93, where the slope of the line is 238:

volume of CO_2 = 238 $\times$ mass of $CaCO_3$, where the slope, with units, is 238 mL CO_2/g $CaCO_3$)

Rearrange to solve for mass of $CaCO_3$:

mass of $CaCO_3$ = volume of CO_2 / 238 mL CO_2 (g $CaCO_3$)$^{-1}$

2. What is the percent $CaCO_3$ in your Tums sample? Use the equation:

%$CaCO_3$ = 100% $\times$ mass of $CaCO_3$/mass of Tums

Part 3. $CaCO_3$ in Tums tablet using mass lost method

1. What is the mass of $CO_2(g)$ produced from the reaction?
2. How many moles of $CO_2(g)$ were produced?
3. How many moles of $CaCO_3(s)$ must have been present to produce this amount of $CO_2(g)$?
4. What mass of $CaCO_3(s)$ must have been present?
5. What is the percent $CaCO_3$ in your Tums sample?
6. How do the results for %$CaCO_3$ from the two methods compare?
7. Which method do you think gives better results? What are possible sources of error in each method?

SUMMARY OF MATERIALS AND CHEMICALS NEEDED FOR CHAPTER 7. PERCENT COMPOSITION.

Equipment required

Item	For 5 pairs	For 10 pairs
Microscale Gas Chemistry Kit (See Chapter 1)	5	10
top-loading balance	2 - 3	3 - 5
analytical balance	2 - 3	3 - 5
disposable pipet (for spatula)	5	10

Materials required

Item	For 5 pairs	For 10 pairs
4 L pail (plastic ice cream pail)	2 - 3	3 – 5
Tums tablets	5	10

Chemicals required

Item	For 5 pairs	For 10 pairs
HCl (3.0 M)	40 cm	80 cm
$CaCO_3$(s)	5 g	10 g