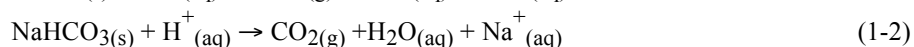
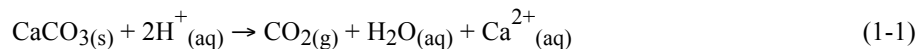


## Determination of % Carbonate in Antacids by Gas Collection

Experiment adapted from Hutton, W., *General Chemistry Laboratory Text*, Ed.2, Charles E. Merrill, Columbus, Ohio (1968) page 57

In this experiment you will be given a sample of a commercial antacid that contains carbonate in one or more various forms, typically calcium carbonate ( $\text{CaCO}_3$ ) or sodium bicarbonate ( $\text{NaHCO}_3$ ). In addition to the active ingredient the antacids contain inert ingredients or binders that allow the product to be easily pressed into a tablet. You will determine the amount of  $\text{CO}_3^{2-}$  in the sample by adding excess  $\text{HCl}$  to convert any  $\text{CO}_3^{2-}/\text{HCO}_3^-$  to  $\text{CO}_2$  gas. Since the binders will not react with  $\text{HCl}$  to form a gas, the analysis will be selective for carbonate compounds. Representative net ionic equations for the carbonate reactions are as follows:



By collecting the gas in a system with a fixed, known volume and measuring the temperature and pressure of the gas, the ideal gas law can be used to determine the moles of  $\text{CO}_2$ . Recall the ideal gas law is as follows:

$$PV = nRT \quad (1-3)$$

P = pressure of  $\text{CO}_2$  in atmospheres

V = volume in liters

T = temperature in Kelvin, ( $0.0^\circ\text{C} = 273.2 \text{ K}$ )

R =  $0.08206 \text{ L atm mol}^{-1} \text{ K}^{-1}$

n = moles of  $\text{CO}_2$  generated

Since you will not know the chemical form of the carbonate, your results will be reported as percent carbonate ( $\text{CO}_3^{2-}$ , fw = 60.01 grams/mole). Once the percent carbonate is known it is a straightforward process to calculate either the percent  $\text{CaCO}_3$  or  $\text{NaHCO}_3$  in the antacid.

A diagram of the system you will be using is given in Figure 1-1.

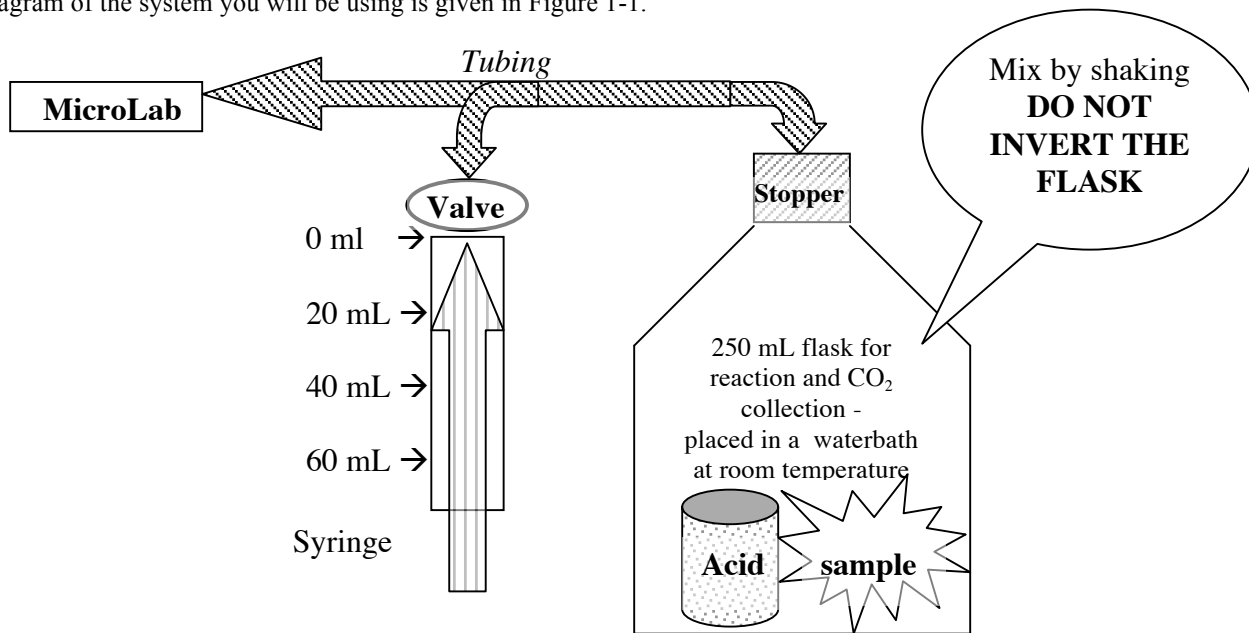


Figure 1-1

## 1-2

A weighed sample of antacid is placed at the bottom of the 250 mL sidearm reaction flask. A plastic vial containing excess hydrochloric acid is carefully lowered into the flask with a string so as not to spill acid onto the sample. A rubber stopper is firmly seated in the top of the flask and the entire flask is placed in the constant temperature bath. With the system at constant temperature, the vial of acid is tipped so the contents will come into contact with the antacid sample and generate  $\text{CO}_2$ . The formation of gas in the closed system will cause an increase in pressure that will be measured by the MicroLab interface and recorded by the computer. The increase in pressure is directly related to the moles of  $\text{CO}_2$  gas that are formed according to equation 1-3. In turn, the moles of  $\text{CO}_2$  are related to the carbonate in the sample by equations 1-1 and 1-2.

Both the pressure and temperature needed for equation 1-3 will be measured directly through the interface. The volume  $V$  in equation 1-3 is the entire volume of the gas collection system, which includes the connecting tubing and the “uncalibrated” volume of the 250 mL sidearm flask. This system volume,  $V_o$ , is not easily determined using geometry ( $l \times w \times h$ ) or by filling the system with liquid and accurately measuring the liquid volume with a graduated cylinder. However, by taking advantage of the gas laws that relate pressure and volume for a system that contains a fixed amount of gas, the volume  $V_o$  for the gas collection system is readily obtained.

Compare the diagram in Figure 1-1 to the actual gas collection system at your lab station. Identify the “tee” that connects the reaction flask, the interface and a one-way valve connected to a syringe. When the valve is in the closed position the total volume of the gas collection system will be referred to as  $V_o$ . This will be the status of the system during the gas collection experiments.

If the valve is opened, but the syringe is set to 0 mL, the volume of the system is still  $V_o$ . The pressure of the system under these settings will be identified as  $P_o$ , which for all practical purposes is the atmospheric pressure. If the syringe is now moved back to some volume  $V_x$  (i.e. to the 20 mL mark), with the valve open, the volume of the system becomes  $(V_o + V_x)$  with a lower pressure of  $P_x$ . Since the moles of gas contained in the system and the temperature are constant, the gas law states

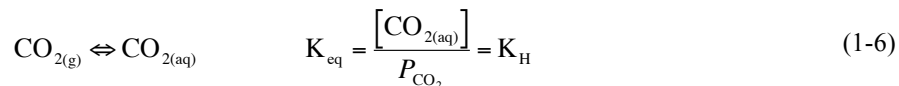
$$P_o \times V_o = P_x \times (V_o + V_x) \quad (1-4)$$

This relationship will hold for any volume that is selected for the syringe, such that a linear relationship of the general form  $y = mx + b$  can be obtained where  $y$  is a pressure ratio and  $x$  is the volume setting of the syringe.

$$\left( \frac{P_o - P_x}{P_x} \right) = \frac{1}{V_o} \times V_x + 0 \quad (1-5)$$

The inverse slope for a plot of equation 1-5 will give the total volume of the system  $V_o$  with the valve closed, which will be the position of the valve when  $\text{CO}_2$  is generated in the reaction vessel. It is this volume that is used in equation 1-3.

An additional consideration in this determination is that the ideal gas law takes into account only the gas phase  $\text{CO}_2$ , while some of the  $\text{CO}_2$  that is generated will dissolve in the solution of acid used to dissolve the carbonate. The amount of dissolved  $\text{CO}_2$  will depend upon the partial pressure of  $\text{CO}_2$  in the system. Since the increase in pressure in the system will only be due to  $\text{CO}_2$  generation, the increase in the total pressure of the system will be equivalent to the partial pressure of the  $\text{CO}_2$ . The equilibrium between the gas phase and dissolved  $\text{CO}_2$  is as follows:



where  $K_{\text{H}}$  is the Henry's Law constant that has units of mol/L/atm. The value of  $K_{\text{H}}$  depends on the gas, the solute and the temperature. The temperature dependence for  $\text{CO}_2$  in water is listed in Table 3.1 for solutions that are 0.2 M NaCl. Since the ionic strength (the total ion concentration in solution) has only a slight impact on  $K_{\text{H}}$  relative to the impact of the temperature, the data in Table 3.1 may be used for this experiment.

Thus, the total moles of carbonate in the antacid sample will be equal to the moles of  $\text{CO}_2$  in the gas phase plus the moles of  $\text{CO}_2$  dissolved in solution. Note that the gas phase volume  $V_o$  will be determined with 20 mL of water in the system, such that the measured value of  $V_o$  will accurately reflect the volume of actually occupied by the gas phase  $\text{CO}_2$ .

**Table 1-1 Henry's Law Constants for CO<sub>2</sub> at Selected Temperatures**Source: <http://lawr.ucdavis.edu/classes/ssc102/Section5.pdf>

Temp K	K <sub>H</sub> (mol/L/atm)
273	0.073
278	0.061
283	0.051
288	0.043
293	0.037
298	0.033
308	0.026

**Objectives:**

1. To determine the percent carbonate by a gas collection technique.
2. To learn techniques for generating and collecting gases, and for measuring gas volumes and pressures.

**Apparatus:** Gas collection apparatus consisting of a 250 mL side arm flask with connecting hose, 25 mL plastic vial with 10 cm of thread tied to the top, no. 6 solid rubber stopper, water bath at room temperature, 2 utility clamps, 1 ring stand, 60 mL plastic syringe connected to a one way miniature luer lock valve, MicroLab interface with a model 103 thermistor and pressure sensor, analytical balance with 0.1 mg precision, stainless steel micro-spatula.

**Chemicals:** Commercial antacids, 1 M HCl, water at room temperature.

**Safety, Environmental, and Economic Concerns:**

1. 1 M HCl is irritating to the skin and hazardous to the eye. Take caution in using this chemical. If your skin or clothing comes in contact with 1 M HCl, rinse the affected area with water. Wipe up spills with a wet sponge and then rinse the sponge in flowing water. If large quantities of the acid spill, contact your instructor immediately. It is imperative that you wear safety glasses when working with 1 M HCl.
2. Solutions used in this experiment may be discarded down the drain with copious amounts of water.

**Notes on Experimental Procedure:**

1. The pressure transducer on the interface can measure up to 2 atm (1520 torr). The amounts of antacid suggested in the experiment are designed to keep the pressure of the system well within these limits. Do not exceed the suggest amounts of sample.
2. When adding the antacid to the side arm flask, avoid have the sample stick to the side of the flask.
3. When weighing the sample, be sure to remove the plastic weigh boat from the balance when adding or removing material. Do not add or remove any substance to a container that is on the balance pan.
4. The HCl will be in a pump dispenser on the reagent bench.

## Experimental Procedure:

### Set up the measurement system:

Two people may work together in this experiment. Only one set-up (Figure 1-1) will be needed. Each person will record all observations and results in their own lab notebook and will complete their own report.

Each station should have a water bath of room temperature water. If not, fill the container with water that is about room temperature. While it takes a long time for the volume of water in the water bath to come to room temperature, if the initial temperature is close to that of the room only a small temperature change will be observed over the course of any one given experimental run.

Set-up the Microlab interface for 3 sensors (see Appendix F). The **thermistor** will be calibrated with model 103 factory calibration. The **pressure** sensor will use the factory calibration in torr. The **time** can have units of seconds. After the sensors have been selected, place time on the graph x-axis and in column A of the spreadsheet. Place pressure on the left hand y-axis and in column B of the spreadsheet. Place temperature on the right hand y-axis and in column C of the spreadsheet. Place all three sensors in the display window. When placed in the display the pressure will be about 740 – 750 torr and the temperature will be about 22°C. If this is not the case, consult your instructor, Set the repeat interval to 5 seconds.

Check the way in which the syringe connects to the one-way valve. At this point the syringe should not be attached. The tubing from the tee should also be connected to the back of the interface, which is where the input for the pressure sensor is located. The thermistor should be in the water bath, but off to one side so it will be easy to place the side arm flask into the water bath.

### Determination of system volume:

Using a graduated cylinder, place 20 mL of water in the plastic vial that will be used to contain the acid. Lower this vial into the flask and drop the string into the flask. Since the vial and 20 mL of liquid will be in the system when the CO<sub>2</sub> is actually generated, having it present at this time gives the correct value of the gas volume in the system, V<sub>0</sub>. Firmly place the rubber stopper in the flask. Clamp the flask in the water bath so that it is immersed up to about the bottom of the neck. Open the stopcock so that the system is at atmospheric pressure (the syringe should not be connected). At this point you can start the data collection, which will begin recording the baseline temperature and pressure (check that the system is set for data collection at 5 second intervals).

With the plunger of the syringe set to 0 ml, connect the syringe to the stopcock. You should not observe any change in the pressure as a result of this. Allow data collection to continue for another minute before proceeding (do not stop the data collection however).

Pull the syringe plunger back to the 20 mL mark and hold it in that position while you observe the pressure in the spreadsheet. An easy way to keep the plunger in a fixed position is to apply pressure to the shaft of the plunger with your thumb, rather than grasping the end of the plunger and trying to keep it at one position. As soon as you have 3 or 4 pressure measurements that are constant to within about 0.5 torr, pull the plunger back to 40 mL and again wait for 3 or 4 successive pressure readings to agree to within 0.5 torr. Repeat this again for 60 mL. After the readings for 60 mL are obtained, return the plunger back to 0 mL and collect data for about one more minute and then stop the data collection. Save this file. Print the graph (right click in the graph window and select print) and on it write the average pressure at each of the volume settings (list the volume as well). To get the average pressure for a set of points from the spreadsheet data, refer to page F-2, the first paragraph under data analysis. Enter this data in a table in your notebook. Include the pressure at both the initial 0 setting and the final 0 setting. In Excel, use the volume and pressure data to plot equation 1-5 and report the volume V<sub>0</sub> of the system (see model spreadsheet on page 1-7 of this experiment).

Leave the side arm flask in the water bath. Disconnect the syringe from the valve. The valve should be open. Start the data collection program. With plunger at 0 mL reconnect it to the valve and repeat the process from the previous paragraph to obtain a second value of V<sub>0</sub>. The two values should agree within 3% to 4%.

After the system volume has been determined, empty the water from the sidearm flask.

**Determination of Henry's Law Constant:**

Obtain a vial containing a sample of pure sodium carbonate,  $\text{Na}_2\text{CO}_3$ , from your instructor. It should contain enough  $\text{Na}_2\text{CO}_3$  for you to weigh out 3 samples of about 0.20 to 0.25 g each. Weigh each sample to the nearest 0.0001 g. To weigh the sample, place a plastic weigh boat on the balance and set the tare to 0. Enter this in your notebook. Remove the weigh boat from the balance and add some  $\text{Na}_2\text{CO}_3$  (about a third of the amount in your vial). Place the weigh boat back on the balance. If you need to add/remove material, take the weigh boat off of the balance pan before doing so. Enter this weight in your notebook. Carefully pour the weighed  $\text{Na}_2\text{CO}_3$  into the flask (see note 2) and reweigh the weigh boat. This will take into account any small amount of material that was not transferred.

Place 20 mL of 1 M HCl from the pump dispenser in the plastic vial with the string around the top. Carefully lower the vial into the flask and set it on the bottom of the flask without spilling any of the contents. Drop the string completely into the flask. Place the rubber stopper firmly in the flask and clamp the flask in the water bath.

If you have not yet done so, remove the syringe from the valve and open the valve so the system is at atmospheric pressure. Start the data collection and record data for about 1 minute with the valve open. Close the valve and continue collecting data for another minute. At the end of this minute, with the valve closed, unclamp the flask and tip the container of acid, swirling the flask so as to completely mix the antacid with the HCl. Clamp the flask back in the water bath and continue data collection for about 10 minutes.

After 1-2 minutes, while still collecting data, unclamp the flask and mix the contents by vigorous shaking for about 10 seconds and then clamp the flask back in the water bath for about 1 minute. **When shaking, do not allow the solution to enter the tubing that connects to the Microlab (ie, don't invert the flask!). If you do get water in this tubing, check with your instructor about the best way to remove the water.** Watch the spreadsheet for an increase in pressure. The  $\text{CO}_2$  tends to build up in the solution and shaking helps achieve equilibrium with the gas phase (equation 1-6). Repeat this shaking about every 60 to 90 seconds. After about 4 or 5 repetitions of mixing/shaking there should be minimal change in the pressure. At this point you can stop the data collection.

After data collection has stopped release the pressure in the system by opening the valve. Remove the flask from the water bath and remove the stopper. Have some paper towels handy to dry the outside of the flask. Take the flask to a sink, rinse the flask with water and then empty the contents into the sink. The plastic vial should be emptied from the flask. Rinse the flask and vial several times.

Save the data file and print a copy of the graph. On the graph indicate the initial and final pressure for the experiment. These pressures, the temperature and mass of sample should all appear in your notebook. Your notebook should also have a sketch of the raw data graph you obtained with some estimate of the scale for each axis.

Repeat this procedure for two more samples of  $\text{Na}_2\text{CO}_3$ . To calculate the Henry's Law constant, assume that all of the  $\text{Na}_2\text{CO}_3$  reacts to produce  $\text{CO}_2$  and that the gaseous form of  $\text{CO}_2$  is contained in your system volume at room temperature and pressure. Calculate the moles of  $\text{CO}_2$  in the gas phase and the partial pressure of that  $\text{CO}_2$  gas. Then determine the remaining moles of  $\text{CO}_2$  that must be dissolved in the solution in the flask. Given that you know the volume of that solution, you can calculate the concentration of  $\text{CO}_2(\text{aq})$  and the resulting Henry's Law constant.

**Determination of carbonate in an antacid:**

Obtain a vial of an antacid from your instructor. The amount you need to weigh out will depend on the type of antacid you are given. The antacid you are given has ID \_\_\_\_\_ and you are to weigh between \_\_\_\_\_ and \_\_\_\_\_ g for a single analysis. To weigh the sample, place a plastic weigh boat on the balance and set the tare to 0. Enter this in your notebook. Remove the weigh boat from the balance and add some antacid. Place the weigh boat back on the balance. If you need to add/remove material, take the weigh boat off of the balance pan before doing so. Enter this weight in your notebook. Carefully pour the antacid into the side arm flask (see note 2) and reweigh the weigh boat. This will take into account any small amount of material that was not transferred.

## 1-6

Place 20 mL of 1 M HCl from the pump dispenser in the plastic vial with the string around the top. Carefully lower the vial into the flask and set it on the bottom of the flask without spilling any of the contents. Drop the string completely into the flask. Place the rubber stopper firmly on the top of the flask and clamp the flask in the water bath.

If you have not yet done so, remove the syringe from the valve and open the valve so the system is at atmospheric pressure. Start the data collection and record data for about 1 minute with the valve open. Close the valve and continue collecting data for another minute. At the end of this minute, with the valve closed, unclamp the flask and tip the container of acid, swirling the flask so as to completely mix the antacid with the HCl. Clamp the flask back in the water bath and continue data collection for about 10 minutes.

After 10 minutes, while still collecting data, unclamp the flask and mix the contents by vigorous shaking for about 10 seconds and then clamp the flask back in the water bath for about 1 minute. Watch the spreadsheet for an increase in pressure. The  $\text{CO}_2$  tends to build up in the solution and shaking helps achieve equilibrium with the gas phase (equation 1-6). Repeat this shaking about every 60 to 90 seconds. After about 4 or 5 repetitions of mixing/shaking there should be minimal change in the pressure. At this point you can stop the data collection.

After data collection has stopped release the pressure in the system by opening the valve. Remove the side arm flask from the water bath and disconnect it from the system tubing using the luer lock fitting closest to the side arm. Have some paper towels handy to dry the outside of the flask. Take the flask to a sink, rinse the flask with water and then empty the contents into the sink. The plastic vial should be emptied from the flask. Rinse the flask and vial several times.

Save the data file and print a copy of the graph. On the graph indicate the initial and final pressure for the experiment. These pressures, the temperature and mass of sample should all appear in your notebook. Your notebook should also have a sketch of the raw data graph you obtained with some estimate of the scale for each axis.

Complete the analysis of at least 3 samples of the antacid.

**Report:** Complete the Excel spreadsheet for this experiment that has been provided via Moodle. You are to do all the calculations within the spreadsheet. The spreadsheet is based on the worksheet on page 1-8 and you may wish to refer to the worksheet as you complete the spreadsheet. Examples of all calculations (with units) need to be in your lab notebook. Data and final results should be entered with appropriate significant figures, but do not round intermediate values used in calculations. Complete a written report that follows the format on page A-9. Comment on what you learned about antacid tablets, factors that affect precision or any of the areas mentioned in the paragraph on results on page A-8. Include the completed worksheet, graphs of Microlab results (with figure numbers and legends), and copies of all notebook pages, including those that contain sample calculations.

## 1-7

### Excel analysis for system volume:

To analyze the pressure and volume data according to eqn 1-5, set-up an excel spreadsheet as given in the figure below. Excel can be opened without closing Microlab. Complete the data analysis as listed in the figure below. Given that the flask is designated as 250 mL, does your volume make sense?

	A	B	C	D
1				
2		syringe mL	$(P_o - P_x) / P_x$	$P_x$ - torr
3		0	$=(\$D\$3 - D3) / D3$	xxx.xx
4		20		
5		40		
6		60		
7		0		
8				
9	Set up the above table with data in Col B and Col D			
10	The pressure in D3 is $P_o$			
11	Enter the formula in C3 as listed			
12	Copy the formula to C4:C7			
13	Highlight B3:C7			
14	Select Chart Wizard icon for XY scatter plot			
15	Complete all steps to generate the plot			
16	Right click on a point, select add trendline			
17	Choose option to display equation			
18	Right click on the equation for the line			
19	Format data labels for scientific display			
20	Inverse of slope is volume of system			
21				
22	You can also use the slope formula			
23			$=\text{SLOPE}(C3:C7, B3:B7)$	
24	take inverse of this value to get system volume			

### Worksheet hints for page 1-8:

- The entry "Gas volume" is the system volume determined above. It is the volume that contains the  $\text{CO}_2$  gas. Note that this volume has already been corrected for the 20 mL of liquid in the system.
- The entry "Solution volume" is the amount of HCl. It is used in conjunction with Henry's Law constant to determine the dissolved  $\text{CO}_2$ . Be sure to use consistent units.
- Use the Henry's Law constant you determined experimentally, assuming it is in the range of 0.025 to 0.045. If your value is outside this range, consult your instructor.

Chemistry 113, Section \_\_\_\_\_

Name \_\_\_\_\_

Instructor \_\_\_\_\_

## Worksheet for the Determination of Percent Carbonate in Antacids by Gas Collection

### System volume

Attach the graphs that show the linear regression for determination of the system volume. The data for this determination should be clearly listed in a table in your lab notes.

	Trial 1	Trial 2	Trial 3
Slope of regression curve	_____	_____	_____
System volume, $V_o$	_____	_____	_____
Average $V_o$	_____		

### Percent carbonate

Sample calculations must be in lab notebook with proper units. Tabulate all results with correct significant figures and units. Do not round intermediate results in calculations.

	Trial 1	Trial 2	Trial 3
Sample mass	_____	_____	_____
Initial pressure	_____	_____	_____
Final pressure	_____	_____	_____
Net pressure $\text{CO}_2$	_____	_____	_____
Temperature	_____	_____	_____
Gas volume (system volume w/ 20 mL water)	_____	_____	_____
Moles $\text{CO}_{2(g)}$	_____	_____	_____
Solution volume	_____	_____	_____
$K_H$	_____	_____	_____
Moles $\text{CO}_{2(aq)}$	_____	_____	_____
Total moles $\text{CO}_2$ formed	_____	_____	_____
Moles carbonate, $\text{CO}_3^{2-}$	_____	_____	_____
Mass carbonate, $\text{CO}_3^{2-}$	_____	_____	_____
Percent carbonate, $\text{CO}_3^{2-}$	_____	_____	_____
Average percent carbonate, $\text{CO}_3^{2-}$	_____		
Standard deviation	_____	(from spreadsheet or calculator, no need for example calcs)	
Relative standard deviation (%)	_____	(standard deviation divided by average % x 100)	
Results as percent $\text{CaCO}_3$	_____	(based on average percent carbonate)	
Results as percent $\text{NaHCO}_3$	_____	(based on average percent carbonate)	