Lab 7: Back Titration of an Antacid

Introduction

When you suffer from heartburn, some of your stomach acid works its way up into your esophagus, causing that burning sensation in your throat and back of your mouth. It seems as if every pharmaceutical company declares that their antacid product can neutralize more acid, provide the longest lasting relief, or work faster than all the rest. Which claims are accurate and which are exaggerations? In order to determine the effectiveness of an over-the-counter antacid you will measure the relative amount of simulated stomach acid neutralized by a tablet.

The most common mode of action that an antacid employs is to remove acid by a neutralization reaction. These antacids are the fast acting type that you will take when you are already experiencing heartburn. The antacid you are testing this week contains the weak base, calcium carbonate CaCO₃. The neutralization reaction can be seen below; note the carbon dioxide gas product, which can explain why some individuals burp or feel bloated after taking an antacid:

 $CaCO_{3(s)} + 2HCI \rightarrow H_2O_{(l)} + CO_{2(g)} + CaCI_{2(aq)}$ (Antacid) + (Acid)

The Experiment

For this experiment, you will measure the relative effectiveness of commercial antacid at the neutralizing stomach acid. The parietal cells (the acid secreting cells) in the stomach secrete hydrochloric acid at a concentration of about 0.155 M; to simulate this stomach acid, we will use 0.15 M HCl in this lab.

A technique called **back titration** is used to determine how much stomach acid is neutralized. Back titration is different from the direct titrations we have used up to this point. In a perfect world, we would run a direct titration to determine the effectiveness of an antacid. This could be accomplished by setting up a titration with the antacid (base) in the flask at the bottom and HCI (acid) in the buret, then titrate until you reached end point. Unfortunately, this is not possible as the time for a complete reaction between the acid and the active ingredient in an antacid can be slow.

In order to complete a titration with slower reacting components such as these antacids, we will perform a back titration. We will add an excess amount of strong acid to the tablet, heat the sample, and give it plenty of time to react completely. After that, we test how much of the strong acid is leftover by titrating against a strong base. This strong acid/base reaction is very quick, so the titration can be performed normally. Based on the amount of strong acid that was added initially and how much acid is left, we will be able to determine how much acid reacted with the antacid. Although the back titration method requires a few additional calculations, it is much more precise and saves a significant amount of time.

Since the goal of this experiment is simply to determine how much acid is neutralized by an antacid, simple ratios are used in the calculations. This way neither the simulated stomach acid nor the NaOH solutions being used for the titration have to be standardized.

The other ingredients should be removed from the neutralized antacid in order to obtain accurate results from the titration. However, it is nearly impossible to recover all of the solution used in an extraction of this type. Even if it were possible, titration of the total volume of acid used would provide a more certain analysis. Therefore, in order to perform a reasonable analysis, it is necessary to establish a volume/mass method that is commonly used in analytical labs that involve the use of ratios. For example, if an antacid tablet was dissolved and diluted in water to 100 mL, 10 mL of that solution could be said to represent 1/10th of the tablet being neutralized. Extensive testing has validated this technique. You applied a similar concept when analyzing the aspirin tablet in Lab 2.

For today's experiment, you will first determine and record the weight of an antacid tablet. Then the tablet is crushed (to speed up the reaction), and the entire amount is placed into a weigh boat or onto a piece of weighing paper, and weighed again. (It is common to lose some the tablet during the crushing.) The crushed tablet is then placed into an exactly measured volume of simulated stomach acid. Crushing the tablet invariably introduces a static electric charge into the powder and it is nearly impossible to transfer the entire crushed tablet into the beaker, so the paper or weigh boat is *reweighed after the transfer* so the exact amount added is known. The solution is gently heated to remove any CO_2 that might remain from the neutralization of any carbonate present. Otherwise, the dissolved CO_2 would act like a buffer during the titration. The neutralized solution is filtered and measured volumes of this solution are titrated. Again, this is more easily demonstrated by working a sample problem.

Example Problem

A student analyzing an antacid tablet determines the mass of the tablet to be 5.216 g. The tablet was placed in a paper towel and crushed. The powder and fragments of the tablet were collected in a weigh boat and the mass of the boat and tablet pieces was determined to be 6.944 g. The tablet was poured into a beaker containing exactly 200.0 mL of 0.15M HCl. The mass of the weigh boat after the transfer was determined to be 2.111 g. The solution was stirred, heated gently for 5 minutes, and then filtered. It was determined that it took exactly 9.45 mL of NaOH solution to titrate a 25.0 mL sample. It took 26.45 mL of NaOH solution to titrate 25.0 mL sample of the original simulated stomach acid. How much simulated stomach acid would the original antacid tablet neutralized in total?

Important data collected

Mass of original antacid tablet	5.216g	
Mass of tablet fragments and weigh boat	6.944g	
Mass of weigh boat following transfer	2.111g	

Mass of tablet powder and fragments used	<u>4.833g</u>
Total volume of simulated stomach acid used	200.0mL
Volume NaOH to titrate 25.0 mL of acid	26.45mL
Volume NaOH to titrate 25.0 mL of neutralized acid	9.45mL

There are several approaches to the solution of this problem. The easiest is to start with the determination of exactly how much of the stomach acid was neutralized in the 25.0 mL sample. This is accomplished by using a ratio. It is known that 26.45 mL of the NaOH solution was needed to reach the endpoint in 25.0 mL of the initial acid and only 9.45 mL of NaOH solution to reach the end point in 25.0 mL of the antacid treated sample.

25.0 mL HCl	x mL HCl	
$\overline{26.45 mL NaOH}$ =	9.45 mL NaOH	

X = 8.93 mL HCl remaining in the sample

Therefore

25.0mL of HCl initial – 8.93mL HCl remaining = 16.07 mL HCl neutralized

However, as this 25.0 mL sample was only a portion of the (1/8) of the total 200.0 mL sample in which the tablet was placed, we need to multiply by 8 to determine the total volume HCl neutralized by the fragments added. So 8×16.07 mL = 128.6 mL HCl neutralized out of the 200mL.

Unfortunately, there is one more necessary step. Remember that we needed to crush the tablet and not all of the original tablet was transferred into the 200mL of simulate stomach acid. The tablet had a mass of 5.216g and we calculated only 4.833g made it into the acid solution. Therefore, to find the total volume of acid that can be neutralized by the whole tablet we have to use the ratio method again.

$$\frac{128.6 \text{ mL HCl}}{4.833 \text{ g tablet}} = \frac{x \text{ mL HCl}}{5.216 \text{ g tablet}}$$

X = 138.8 mL HCl neutralized by whole tablet.

Procedure

Part I: Preparation of Equipment

- 1) Obtain ~300 mL of 0.15M HCI (Simulated stomach acid), and ~100mL of 0.15 M NaOH.
- 2) Rinse your buret 2x with 5-10 mL of the NaOH solution, and then fill to just below the 0.00 mL mark.
- 3) Drain some solution through the tip to make certain there are no air bubbles.
- 4) Record this initial buret volume in your lab book.

Part II: The Neutralization of the Tablet

- 1) Obtain a 400 mL beaker and add 200.0 mL of simulated stomach acid using the 100 mL graduated cylinder.
- 2) Obtain one commercial antacid tablet.
- 3) Weigh and **record the mass** value of the tablet.
- 4) Place the tablet between two paper towels and crush the tablet.
- 5) Transfer as much of the crushed tablet as you can into a weight boat.
- 6) Weigh the tablet and weigh boat together and **record this value**.
- 7) Pour the entire contents of the boat into the 200 mL of HCl.
- 8) Reweigh the boat and **record the value** in your notebook.
 - a. The difference is the amount added to the acid solution.
- 9) Place the beaker onto the hot plate and warm with gentle swirling. The beaker just needs to be warm (not hot).
- 10) Heat for \sim 5 mins and then set aside for part IV.

Part III: Titration of the Simulated Stomach Acid

- 1) Using a graduated cylinder measure out exactly 25.0 mL of simulated stomach acid into a 250 mL Erlenmeyer flask. Repeat with a second flask.
- 2) Add 4-5 drops of Phenolphthalein to each of the two 250 mL Erlenmeyer flask that contain the original simulated stomach acid.
- 3) Using your buret filled with NaOH, titrate the HCI solutions until you reach the endpoint.
 - a. Be sure you are recording the exact initial and final buret readings for each trial.
 - b. Also, make sure have enough solution in your buret before starting the second titration. For example, if it took you 24+ mL to titrate the first sample and you only have 20 mL left in you buret you will not be able to reach the endpoint during the second trial!
- 4) The second trial should agree to within 0.5mL of the total volume NaOH added as compared to the first trial. If it doesn't, you will need to run a third trial to determine which two measurements are correct.
- 5) Average the two closest trials.

Part IV: Preparation and Titration of the Neutralized Stomach Acid Solution

- 1) Obtain a glass funnel and a piece of coarse filter paper.
- 2) Fold the filter paper and place it into the funnel.
- 3) Set up the gravity filtration so the funnel will not tip or fall over as you filter the neutralized stomach acid.
- 4) Gradually pour the partially neutralized acid from Part II through the filter paper, adding additional portions as the level decreases.
 - a. Be careful not to allow the liquid level to raise higher than the level of the paper.
- 5) Filter and collect ~ 90mL of the solution.
 - a. You do not need to filter all 200 mL of solution.
- 6) Use a small amount (~5mL) of your filtrate to rinse your 100 mL graduated cylinder.
- 7) Transfer exactly 25.0 mL of the filtered solution into a 250 mL Erlenmeyer flask. Repeat with a second flask.

- 8) Add 4-5 drops of Phenolphthalein to each of the two 250 mL Erlenmeyer flasks.
- 9) Refill the buret with NaOH solution and record the initial starting volume.
- 10) Titrate the first flask with NaOH until you just reach the endpoint.
- 11) Record the final buret reading.
- 12) Refill the buret with NaOH (if needed) and titrate the second flask .
 - a. Be sure to record the initial and final buret readings!
- 13) The two titrations should agree to with a total of **0.5mL**; if not run a third titration.
- 14) Average the two closest titrations.

Report Sheet

<u>Please ensure that you document all of the values in **black** on your lab notebook page!</u> These are numbers that you will enter into LabFlow's Data Report Sheet, and will be used to calculate the values in **red**.

Part II:		
1. Mass of Whole Tablet (g)		
2. Mass of Crushed Tablet and Boat (g)		
3. Mass of Boat after Tablet removed (g)		
4. Mass of Tablet added to 200mL acid (g)		
Part III:	Trial 1	Trial 2
5. Stomach Acid (HCl) used (mL)		
6. Initial Buret Reading (mL)		
7. Final Buret Reading (mL)		
8. Volume NaOH added (mL)		
9. Average Volume NaOH Used (mL)		
Part IV:	Trial 1	Trial 2
10. Volume filtered acid added to flask (mL)		
11. Initial Buret Reading (NaOH) (mL)		
12. Final Buret Reading (NaOH) (mL)		
13. Volume NaOH Added (mL)		
14. Volume HCl Remaining in sample (mL)		
15. Average Volume HCl Remaining in sample (mL)		
16. Volume HCl Neutralized in 25 mL Sample (mL)		
17. Volume HCl Neutralized in 200 mL Solution (mL)		
18. Volume HCl Neutralized by Whole Tablet(mL)		