13.1 Titration - Pre-Lab Questions

Name: ____ Date: __________________

Instructor: ____ Section/Group: ____________

1. Calculate the maximum volume in mL of 0.15 M HCl that each of the following antacid formulations would be expected to neutralize. Assume complete neutralization. (Show work.)
    a. A tablet containing 250 mg $Al(OH)_3$ and 150 mg $Mg(OH)_2$

    b. A tablet containing 850 mg of $CaCO_3$

2. A student is given an antacid tablet that weighed 5.6832 g. The tablet was crushed and 4.3628 g of the antacid was added to 200 mL of simulated stomach acid. This was allowed to react and then filtered. It was found that 25.00 mL of this partially neutralized stomach acid required 8.50 mL of a NaOH solution to titrate it to a methyl red endpoint. It took 21.54 mL of this NaOH solution to neutralize 25.00 mL of the original stomach acid.

    a. How much of the “stomach acid” had been neutralized in the 25.00 mL sample that was titrated? (Show work.)

    b. How much stomach acid was neutralized by the 4.3628 g sample used? (Show work.)

    c. How much stomach acid would have been neutralized by the original 5.6832 g tablet? (Show work.)

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13.2 Titration - Introduction

Every day we are bombarded by commercials dealing with acid indigestion. Each company declares their product can neutralize more acid, provide the longest-lasting relief, or work faster than all the rest. Some products actually "take up to four (4) days to provide complete relief" and yet they claim instant effectiveness. Which claims are accurate and which are exaggerations? That is what this experiment is designed to discover. In order to determine the effectiveness of a variety of over-the-counter antacids, you will measure the relative amount of simulated stomach acid neutralized by each.

Relevance

Most antacid products contain one or more of four active ingredients: aluminum hydroxide, magnesium hydroxide, calcium carbonate, and sodium bicarbonate. One other product contains bismuth subsalicylate. Although all of these products provide relief from acid indigestion in a minute or less – they all vary in how long they maintain this relief – from about 10 minutes to more than 1.5 hours. Antacid sales amounted to more than 10 billion dollars in 2010. This revenue guarantees that development of safe, fast-acting, long-lasting antacids will continue to be a focus at many pharmaceutical companies around the world. As a student in chemistry, someone who may be looking for a job at these pharmaceutical companies some day, knowledge of the chemical efficacy of antacids may just provide the background you need to start your career.

> ***The analytical skills you learn and employ in this experiment may help you launch successful careers within the chemical/pharmaceutical/medical industries. These are great skills that chemists use every day.***

Background

The over-the-counter (OTC) antacids fall into two categories: those that neutralize acid and those that prevent the production of stomach acid. (The latter are called H_2 blockers. Since H_2 blockers act within the body on various enzymes, their testing is beyond the scope of this lab.) The more common antacids that neutralize stomach acid after it has already been formed will be the focus of this experiment.

As mentioned in the previous section, there are a number of active ingredients used in the different OTC antacids. Each ingredient has a slightly different mechanism of reaction and slightly different side effects.

Aluminum and Magnesium Salts

Antacids that contain both magnesium and aluminum hydroxides were once thought to be ideal. Aluminum hydroxide dissolves slowly in the stomach and works gradually, providing long-lasting relief. However, trivalent ions like aluminum (Al^{3+}) tend to cause constipation. Magnesium hydroxide (and/or magnesium carbonate) dissolves rapidly and neutralizes acids effectively. Divalent ions tend to have a laxative effect. Some antacids contain only aluminum hydroxide and others contain only magnesium hydroxide. Antacids that contain both aluminum and magnesium hydroxides provide quick, long-lasting relief with less risk of diarrhea or constipation. There has been some recent concern that long-term (chronic) use of aluminum containing antacids may weaken bones by depleting the body of phosphorus and calcium.

Calcium Carbonate

Calcium carbonate (chalk, limestone, marble, coral reef, etc.) has been used as an antacid for more than 2,000 years. Calcium carbonate acts fast and neutralizes acids for a relatively long time. It is also an inexpensive source of calcium; amounts of available calcium per tablet/dose range from ~500 mg to 1,000 mg. Because of the recent push to add calcium to the diet, many antacids have added calcium carbonate to their formulations so that they can claim to be a source of dietary calcium.

Sodium Bicarbonate

Sodium bicarbonate, baking soda, is the most readily available of all the antacids. It neutralizes acids quickly. The common sign that the baking soda is working is the release of CO_2 accompanied by a (usually impressive) burp. Although baking soda provides a short-term solution to indigestion, too much bicarbonate can alter the body’s acid-base balance and lead to serious, perhaps fatal, metabolic imbalance, alkalosis. Individuals who must limit their sodium intake should use a different antacid if at all possible.

Other Ingredients

A variety of other ingredients are commonly found in antacids. These extra ingredients include flavors, a sweetener (artificial or natural), usually a dye (for color), usually cornstarch, possibly a fragrance, and generally a binder. The flavors, sweetener (sucrose or dextrose or one of several artificial sweeteners) and fragrance (if any) are designed to make the antacid more palatable. Cornstarch is primarily added to keep the tablet from sticking in the “pill press” where the tablets are formed. Tablets also contain a binder-like talc, attapulgite, bentonite, or kaolin. These are clay-like minerals that when compressed hold their form. These are used to “bind” the ingredients into a solid tablet that is sturdy enough to survive handling. They also act as anti-caking agents that absorb water and prevent the antacid from degrading.

The Antacids

In this experiment, each group will test a different type of commercial antacid tablet and the results of each group will be compared. The tablets will be analyzed only for their ability to neutralize acids. Specifically the quantity of simulated stomach acid neutralized by each antacid will be determined. The base in the antacids varies with the brand. The table below lists several commercial brands and their active agent(s).

Brand	**Active Agents(s)**
Alka-Seltzer	$NaHCO_3$ and/or $KHCO_3$
Equate	$Al(OH)_3$ and $Mg(OH)_2$
Gaviscon	$Al(OH)_3$
Maalox (liquid)	$Al(OH)_3$ and $Mg(OH)_2$
Maalox (tablet)	$CaCO_3$

Milk of Magnesia	$Mg(OH)_2$
Pepto-Bismol	HOC_6H_4COO
Pepto-Bismol Children's	$CaCO_3$
Rolaids	$CaCO_3$ and $Mg(OH)_2$
TUMS	$CaCO_3$

The neutralization of acids by these bases is outlined below. Magnesium hydroxide is a strong but insoluble base; it is neutralized completely in stomach acid.

$$Mg(OH)_{2(s)} + 2\ H^+_{(aq)} \rightarrow Mg^{2+}_{(aq)} + 2\ H_2O_{(l)}$$

The neutralization of carbonate and hydrogen carbonate follows the same stepwise sequence. Magnesium or calcium carbonate is dissolved by acid to liberate the metal ion and form hydrogen carbonate ion (bicarbonate ion). The bicarbonate ion then reacts with additional acid to form the “intermediate” carbonic acid that decomposes into carbon dioxide and water.

$$CaCO_{3(s)} + H^+_{(aq)} \rightarrow Ca^{2+}_{(aq)} + HCO^-_{(aq)}$$

$$HCO_3^-{}_{(aq)} + H^+_{(aq)} \rightarrow H_2CO_{3(aq)} \rightarrow CO_2(g) + H_2O_{(l)}$$

Bismuth subsalicylate is found in adult Pepto-Bismol; in addition to its antacid affect, it also exhibits anti-diarrheal and mild antibiotic activity.

$$BiO(HOC_6H_4COO)_{(s)} + 3\ H^+_{(aq)} \rightarrow Bi^{3+}(aq) + H_2O_{(l)} + HOC_6H_4COOH_{(s)}$$

Aluminum hydroxide is not as straightforward in its antacid abilities. Each hydroxide ion is removed in sequence as expected; however, only two (2) of the three (3) available hydroxides are generally utilized for stomach acid neutralization. Removal of the final hydroxide ion usually does not occur unless the pH is below 4. This does make aluminum hydroxide a much longer-acting antacid, but its acid neutralizing capabilities are seldom fully used.

$$Al(OH)_{3\ (s)} + H^+_{(aq)} \longrightarrow Al(OH)_2^+{}_{(aq)} + H_2O_{(l)}$$

$$Al(OH)_2^+{}_{(aq)} + H^+_{(aq)} \longrightarrow Al(OH)^{2+}_{(aq)} + H_2O_{(l)}$$

$$Al(OH)^{2+}_{(aq)} + H^+_{(aq)} \xrightarrow{pH<4} Al^{3+}_{(aq)} + H_2O_{(l)}$$

The Experiment

For this experiment, you will measure the relative effectiveness of how well a commercial antacid neutralizes 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.

A technique called **back titration** is used to determine how much stomach acid is neutralized. Since the goal of this experiment is simply to determine "how much acid is neutralized" by an antacid and then compare this to other brands of antacids, simple ratios are used in the calculations. This way neither the simulated stomach acid nor the NaOH solution being used for the titration has to be standardized.

The other ingredients must be removed from the neutralized antacid in order to get good results from the titration. 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 allow only a single determination; analysis of several small samples of this solution would provide a more certain analysis. In order to do this, it is necessary to establish a volume/mass relationship between the solution and the tablet used in its neutralization. There is a reliable method that is commonly used in analytical labs that involves the use of ratios. For example, if an antacid tablet was dissolved and diluted by 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.

For today's experiment, you first determine and record the weight of an antacid tablet. Then the tablet is crushed (to speed up the reaction), the entire amount placed into a weigh boat or onto a piece of weighing paper, and weighed again. (It is common to lose some of 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 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 chooses an antacid tablet and determines its mass to be 5.2163 g. The tablet is 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 6.9438 g. The tablet was poured into a beaker containing exactly 200.00 mL of 0.15 M HCl. The mass of the weigh boat after the transfer was determined to be 2.1106 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 25.00 mL of this solution to a methyl red endpoint. It took 26.45 mL of this NaOH solution to titrate a 25.00 mL sample of the original simulated stomach acid. How much simulated stomach acid would the original antacid tablet have neutralized?

Important Data Collected	
Mass of original antacid tablet	5.2163 g
Mass of tablet fragments and weighing boat	6.9438 g
Mass of weighing boat following transfer	2.1106 g
(Mass of tablet powder and fragments used)	(4.8332 g)

Total volume of simulated stomach acid used	200.00 mL
Vol. NaOH to titrate 25.00 mL of original	26.45 mL
Vol. NaOH to titrate 25.00 mL of neutralized solution	9.45 mL

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. This is most easily accomplished by using a ratio. It is known that 26.45 mL of the NaOH solution was needed to neutralize 25.00 mL of the initial acid. Use this to determine how much acid was "left" in an equal quantity of the antacid neutralized sample. ***Remember, you are only trying to determine how much acid was neutralized***. In advertising, this is referred to as the ***volume*** of stomach acid neutralized.

$$\frac{25.00 \text{ mL HCl}}{26.45 \text{ mL NaOH}} = \frac{x \text{ mL HCl}}{9.45 \text{ mL NaOH}}$$

$$x = 8.93 \text{ mL HCl remaining}$$

Therefore

$$25.00 \text{ mL HCl initial} - 8.93 \text{ mL HCl remaining} = 16.07 \text{ mL HCl neutralized}$$

Since the 25.00 mL sample represents one-eighth (1/8) of the total HCl neutralized by tablet, then 8×16.07 mL = 128.56 mL of HCl would be neutralized by the entire sample used.

Remember that not all of the tablet was neutralized and the quantity of acid that would have been neutralized by the total tablet will be greater. There are two approaches that can be used to determine the quantity of acid that would have been neutralized by the total antacid tablet: by ratio and by percentage. Both methods work equally well; the choice of which to use is a matter of personal preference. The ratio method will be shown first.

Ratio method

$$\frac{4.8332 \text{ g tablet used}}{5.2163 \text{ g total wt tablet}} = \frac{128.56 \text{ mL HCl}}{x \text{ mL HCl}}$$

$$x = 138.75 \text{ mL HCl neutralized by tablet}$$

This same result can also be obtained through the use of the fraction of the tablet that was *neutralized*. This fraction or "percentage" method is shown here.

Percent method

$$\frac{4.8332 \text{ g tablet used}}{5.2163 \text{ g total wt tablet}} = .92656 \text{ fraction tablet used}$$

$$\frac{128.56 \text{ mL HCl neutralized}}{.92656 \text{ fraction tablet used}} = 138.75 \text{ mL HCl neutralized by tablet}$$

13.3 Titration - Procedure

Safety Notes: Hydrochloric acid and sodium hydroxide are considered corrosive irritants even as dilute solutions. Sodium hydroxide represents a serious eye hazard. Eye protection must be worn during the laboratory. All solutions and waste other than the filter paper may safely be disposed down the drain. The filter paper can be discarded in the trashcan.

Each group must use a different commercial antacid so that the acid neutralizing “strength” of each product can be compared.

Part I: Preparation of Equipment

1. Obtain three (3) 125-mL Erlenmeyer flasks, two (2) 250-mL beakers, two (2) 400-mL beakers, stir bar, and a hot plate/stirrer. You will also need two (2) 50-mL burets, a wash bottle of distilled water, a ring stand, and buret clamp.
2. Use one of the 400-mL beakers to obtain ~300 mL of 0.15 M HCl (***simulated stomach acid***) and one of the 250-mL beakers to obtain ~100 mL of 0.15 M NaOH.
3. Select one of the burets to be used for the NaOH solution. Rinse the buret with a small amount of the NaOH solution. Allow the NaOH to drain through the buret tip (discard down the drain), and then fill the buret with the NaOH solution. Try to make certain that the initial buret reading is 0.00 mL.
4. Rinse each of the 125-mL Erlenmeyer flasks with a small quantity of the HCl solution.
5. Swirl the acid in each flask and then pour this down the drain with plenty of running water.
6. Thoroughly rinse each of the flasks with distilled water to remove any residual HCl that might remain.
7. Allow one of the Erlenmeyer flasks to dry; it will be used for the filtration of your neutralized solution.
8. You do not need to dry the other two (2) Erlenmeyer flasks.
9. Rinse the remaining 400-mL beaker and stirring bar with some of the HCl solution.
10. Carefully swirl the acid solution around the beaker in order to remove any residual base that might be present.
11. Pour the acid down the drain with plenty of running water and then thoroughly rinse the beaker and stir bar with distilled water.

12. ***Dry both the stir bar and the 400-mL beaker*** with a paper towel. It is important that no residual water be on the stir bar or in the beaker. Place the stir bar in this 400-mL beaker.

13. Rinse the remaining buret with a small quantity of the HCl solution. Allow the acid to completely drain from the tip of the buret (discard this HCl) and then fill the buret with fresh HCl. Make certain that the buret is filled exactly to the 0.00 mL mark and that no bubbles are trapped in the tip.

14. Use the HCl-filled buret to transfer exactly 25.00 mL of acid into each of the two 125-mL Erlenmeyer flasks.

15. Refill the HCl buret and make certain the liquid is at the 0.00 mL mark.

16. You will now transfer four 50.00 mL (***4 x 50.00 mL***) volumes of HCl into the rinsed and dried 400-mL beaker, making a **t*otal of 200.00 mL*** of the simulated stomach acid. ***Use a buret for these transfers. Neither a graduated cylinder nor a beaker will be sufficiently accurate (or precise) enough for this transfer.***

17. When you have finished with this measurement, drain all of the remaining acid from the buret and rinse it thoroughly with distilled water. You will need to use this cleaned buret with the neutralized acid solution you will be preparing.

Part II: The Neutralization of the Tablet

1. Obtain one of the commercial antacid tablets (avoid red colored tablets). Inform your instructor the brand of antacid tablet you have chosen.

2. Weigh and record the weight of the tablet.

3. Place the tablet between two paper towels and crush the tablet.

4. Transfer as much of the crushed tablet as you can into a weight boat.

5. Weigh the tablet and weigh boat and record this value.

6. Pour the entire contents of the weigh boat into the 200 mL of HCl. Reweigh the weigh boat and record the value in your notebook.

7. Place the beaker onto the hot plate/stirrer and initiate gentle stirring. Also begin heating (low setting); the beaker just needs to be warm (**not hot**).

8. Allow the beaker to stir for at least 5 minutes.

Part III: Titration of Simulated Stomach Acid

1. Add two drops of methyl red to each of the two 125-mL Erlenmeyer flasks that contain the original simulated stomach acid (0.15 M HCl).

2. Make certain that the NaOH solution is at the 0.00 mL mark in the buret and then begin titrating. Methyl red is red when acidic and becomes orange just before the yellow endpoint.

3. Record the volume of the NaOH at the endpoint and then refill the buret.

4. Repeat this titration with the second flask to the yellow endpoint. This endpoint should be within 0.1 mL of the volume you obtained for the first flask or you will need to repeat this titration.

5. When finished with this part of the titration, discard the contents of the flasks by pouring them down the drain.

6. Rinse each flask thoroughly with distilled water. These will be reused when you titrate the "neutralized solution."

Part IV: Preparation and Titration of Neutralized Stomach Acid

1. Obtain a funnel and a piece of filter paper.

2. Fold the filter paper and place it into the funnel. Place the funnel into the dry Erlenmeyer flask you prepared earlier.

3. Carefully position the **dry** folded filter paper in the funnel and begin pouring the antacid/acid solution into the filter paper.

4. The paper should position itself down in the funnel as the solution dampens the paper. Take care not to tear the paper.

5. Allow the solution to filter into the flask. ***You only need to collect ~75 mL of this solution***.

6. When you have collected 75 mL of filtrate, move the funnel back to the beaker.

7. Use some of this filtrate to rinse the buret that had originally been used for the simulated stomach acid.

8. Allow the filtrate to drain through the stopcock and discard this rinse.

9. Now fill the buret with the filtrate.

10. Transfer exactly 25.00 mL of the filtrate into each of the two 125-mL Erlenmeyer flasks.

11. Add two drops of methyl red indicator to each of the flasks.

12. Make certain that the NaOH buret is filled and record the starting volume.

13. Titrate the first flask with NaOH until you just reach the yellow endpoint.

14. Record the volume of NaOH used.

15. Refill the NaOH buret if necessary, record the starting volume of NaOH and titrate the second flask to the yellow endpoint.

16. If the volume of NaOH required for the two titrations differs by more than 0.1 mL, repeat the titration with a third volume of your neutralized acid.

Your instructor may require you to complete the calculations so that your results can be distributed to the rest of the class at this time.

Clean Up

All solutions may safely disposed of by pouring down the drain followed by rinsing water. Rinse all of the glassware thoroughly with water, dry, and replace in the appropriate storage areas. Rinse each buret with water, allowing the water to drain through the tip, and return to the storage location. Return the ring stand, hot plate/stirrer, and stir bar to their appropriate locations. The funnel may be washed with water, dried, and replace in its cabinet. The filter paper may be safely disposed of in the trash.

13.4 Titration - Data Sheet

Name: ____ Date: ________________

Instructor: ______________________________________ Section/Group: __________

Brand of antacid tablet ______________

Mass of original antacid tablet (g) ______________

Mass of weighing boat and tablet (g) ______________

Mass of weighting boat following transfer (g) ______________

Mass of tablet used(g) ______________

Simulated Stomach Acid	**Trial 1**	**Trial 2**
Volume acid used (mL)		
Initial reading NaOH (mL)		
Final reading NaOH (mL)		
Volume NaOH used (mL)		

Average volume NaOH (mL) ________________

Partially Neutralized Stomach Acid	**Trial 1**	**Trial 2**
Volume acid used (mL)		
Initial reading NaOH (mL)		
Final reading NaOH (mL)		
Volume NaOH used (mL)		

Average volume NaOH (mL) ________________

Class Data

Brand of Antacid	Simulated Stomach Acid Neutralized by One Tablet
Akla Mints	
Alka Seltzer	
Equate	
Gaviscon	
Gaviscon Extra Strength	
HEB Extra Stength	
Maalox	
Maalox Max	
Rolaids	
Rolaids Extra Stength	
TUMS	
TUMS Extra Strength	

Best Value Determination

Brand of Antacid	Cost Per Bottle	Tablets Per Bottle	Cost Per Tablet	Volume of Acid Neutralized	Cost Per mL of Acid
Alka Mints	$1.98	75			
Alka Seltzer	$7.98	72			
Equate	$3.44	100			
Gaviscon	$5.77	100			
Gaviscon Extra Strength	$7.88	100			
HEB Extra Strength	$3.88	100			
Maalox	$5.77	85			
Maalox Max	$5.77	65			
Rolaids	$4.97	100			
Rolaids Extra Strength	$3.49	100			
TUMS	$1.98	96			

TUMS Extra Strength	$3.44	92			

1. Which were the top three antacids in the amount of acid neutralized?

2. What was the best value in terms of cost per mL of volume of acid neutralized?

13.5 Titration - Post-Lab Questions

Name: ____ Date: __________________

Instructor: ____ Section/Group: ____________

1. Why did you have to remove CO_2 from your partially neutralized stomach acid before the titration? (This was done by gently heating the solution.)

2. Why was methyl red used as an indicator in this experiment rather than phenolphthalein?

3. Based upon the data collected by your lab section, are extra-strength antacids more cost effective than the regular strength tablets? (Clearly explain your answer in terms of cost and volume of acid neutralized.)