Experiment 5:
Acid–Base Titrations

Safety First!

Safety goggles must be worn at all times in the laboratory!

Potential Hazards

- HCl (hydrochloric acid) is corrosive, can cause painful chemical burns, and will eat holes in your clothes. Rinse hands with water if you get any on you. See your TA for help cleaning up a spill immediately.
- NaOH (sodium hydroxide) is caustic and can cause chemical burns. These are not painful at first, so you may not realize if you have gotten it on your skin; wash hands after using.
- Indicator solutions are potentially flammable and toxic; keep them away from open flames and do not ingest them.

Waste Disposal

- All waste containing acid and base must be disposed of in the hazardous-waste container.

Experiment Objective(s):

Standardize a solution of base using the analytical technique known as titration.
Measure the quantity of stomach acid that can be neutralized by antacid tablets.

Learning Objectives:

In this experiment you will learn to perform a titration, select suitable indicator, detect an endpoint, and determine the amount of active ingredient in the antacid tablet.

Background:

In preparation for this experiment you should review chapter 4 ,sections 4.5 and 4.7 of your textbook, Chemistry (Chang & Goldsby), Custom Edition for IIT, 2015.
You should also study the technique(s) of titration posted in the Techniques Folder of Blackboard.

Standardization of a Sodium Hydroxide Solution

Titration is the quantitative analysis of the contents of a solution. The solution under study is called the analyte or titrand. The solution that is used to determine the contents of the analyte is called the titrant. In this reaction, the titrant is sodium hydroxide solution, NaOH (aq). For a proper acid-base titration, it is essential to know the concentration of the NaOH (aq) that is required to neutralize the acidic analyte. This concentration can be calculated by accurately weighing a solid sample of sodium hydroxide and dissolving in water to form a known volume of solution. However, it is difficult to accurately weigh sodium hydroxide because it is hygroscopic (meaning that it absorbs water vapor readily from air) forming a partially hydrated solid. Therefore, a solution of NaOH (aq) is usually standardized using an acid known as a

primary standard. A primary standard must be a solid, stable compound that is not hygroscopic, can be easily handled, and is available in very pure form.

For this experiment, you will standardize a solution of NaOH (aq), which has an approximate concentration of 0.3 M, by using potassium hydrogen phthalate, KHP, as a primary standard. KHP has a chemical formula $KHC_8H_4O_4$ and a molecular weight of 204.23 g/mole. When, KHP is dissolved in water to form an aqueous solution, the potassium cation dissociates from the hydrogen phthalate anion according the equation:

$$KHC_8H_4O_4\ (s) \rightarrow K^+\ (aq) + HC_8H_4O_4^-\ (aq)$$

The hydrogen phthalate anion has one acidic proton, which reacts stoichiometrically with one OH^- according to the following equation:

$$OH^-\ (aq) + HC_8H_4O_4^-\ (aq) \rightarrow C_8H_4O_4^{2-}\ (aq) + H_2O\ (l)$$

The previous equation is often written in abbreviated form as

$$OH^-\ (aq) + HP^-\ (aq) \rightarrow P^{2-}\ (aq) + H_2O\ (l)$$

where the letter "P" stands for "phthalate".

For the highest accuracy, a sample size is chosen such that it will consume as large a volume of the base as possible without exceeding the capacity of the buret. At the endpoint, the number of moles of NaOH equals the number of moles of KHP. For example, if a 25 mL buret is used, an amount of KHP is chosen such that it will require approximately 20 mL of 0.3 M NaOH solution to reach the endpoint. Thus, about 0.006 moles, or 1.2 g, of KHP is needed. Keep in mind that you will be using a 50 mL buret in your experiment.

$$\text{M (NaOH)} = \text{M (KHP)} = \frac{\text{moles (KHP)}}{\text{V (NaOH) in liters}} = \frac{\text{g (KHP)}}{204.23\ \text{g/mole}} \times \frac{1000\ mL/L}{mL\ (NaOH)}$$

Once the NaOH solution has been standardized, it can be used to determine the acid content of an analyte.

Determination of the Acid Neutralizing Capacity of an Antacid Tablet

The parietal cells secrete hydrochloric acid, HCl (aq) at a concentration of roughly 0.16 M in the stomach. The flow of HCl (aq) increases when food enters the stomach. If you eat or drink too much, you may develop heartburn or indigestion. Antacids, such as Tums, are used to neutralize this excess acid. The active ingredient in Tums is calcium carbonate, $CaCO_3$, a base. There are

also other ingredients, such as binders present in each tablet. On average, a 1.3 gram tablet contains 0.5 g of calcium carbonate and the rest of the mass consists of inactive ingredients.

HCl (aq) is neutralized by calcium carbonate as illustrated below:

$$CaCO_3\,(s) + H^+\,(aq) \rightarrow Ca^{2+}\,(aq) + HCO_3^-(aq)$$

$$HCO_3^-(aq) + H^+\,(aq) \rightarrow H_2CO_3\,(aq)$$

$$H_2CO_3\,(aq) \rightarrow CO_2\,(g) + H_2O\,(l)$$

To determine the ability of Tums to neutralize acid, we are first going to dissolve the tablet in an excess amount of acid of known concentration. Some of the HCl (aq) will be neutralized by the carbonate and some will remain resulting in an acidic solution. We will then perform a titration with previously standardized NaOH (aq) to determine the amount of excess acid. By knowing the original amount of HCl (aq) added we will subtract the amount remaining to calculate the amount of acid that was neutralized by the Tums tablet and therefore we will know the amount of $CaCO_3$ in the Tums. This method of analysis is called ***back-titration***, where we determine the amount of base by acidifying it with a known quantity of acid and then titrate it ***back*** to neutral.

The reactions above are reversible, which means that carbon dioxide gas, CO_2 (g), dissolved in water will produce some carbonic acid, H_2CO_3 (aq). This acid will react with the NaOH (aq) titrant causing inaccurate results. To avoid this problem, it is important to boil the solution when the carbonate reacts with acid which will remove the carbon dioxide as a gas.

The neutralization reaction of a strong acid, HCl (aq), with a strong base, NaOH (aq), is

$$HCl\,(aq) + NaOH\,(aq) \rightarrow NaCl\,(aq) + H_2O\,(l)$$

When a solution of hydrochloric acid is exactly neutralized with a solution of sodium hydroxide, the number of moles of NaOH consumed will equal to the number of moles of HCl initially present in the analyte. This exact neutralization occurs in a reaction between a strong acid and a strong base at the "equivalence" point; the point at which equivalent amounts of acid and base have been added to the reaction. At this point, the following relationship therefore holds true:

$$(M_{NaOH})(V_{NaOH}\text{ in liters}) = (M_{HCl})(V_{HCl}\text{ in liters})$$

Where M is the concentration in molarity and V is the volume. If three of the above quantities are known, the fourth can be easily calculated.

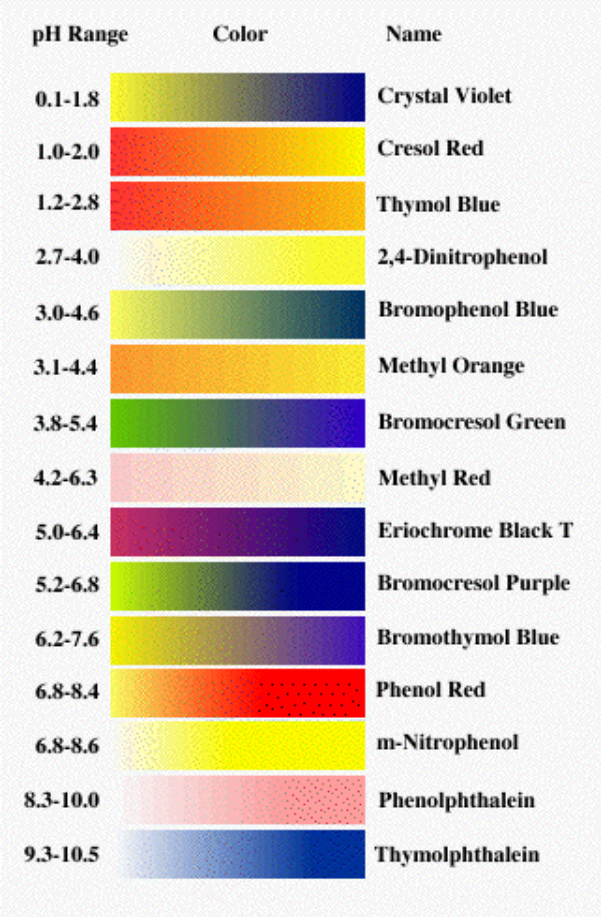
Experiment 5:
Acid–Base Titrations

Indicator Choice

The pH is a scale used to measure acidity and will be discussed in detail next semester. For current experiment, it is only necessary to know that neutral solutions have a pH equal to 7, acidic solutions have pH lower than 7 and basic solutions have a pH greater than 7.

To determine when a solution has been exactly neutralized, an acid-base indicator is used that changes color in a certain pH range. This color change is termed the "endpoint" of the titration; the point at which the titration is determined to be complete. For an accurate titration, the endpoint and the equivalence point must as close as possible. The neutralized solution in our experiment will have a pH equal to 7 and therefore an indicator that changes color near this pH should be used for a strong acid-base titration.

To determine the best indicator for the titration, you are given a chart of various indicators with pH ranges to choose from. *Please see below*



Experiment 5:
Acid–Base Titrations

Procedures:

Equipment and Chemicals

- 250mL Erlenmeyer flasks
- 50mL Buret
- Ring stand
- Clamp
- Glass stirring rod
- Wash bottle of DI water
- Antacid tablets
- 0.3M NaOH (approx.)
- 0.3M HCl (approx.)
- KHP
- Indicator solutions
- Hot plate
- Paraffin film

Follow the procedures outlined on the following pages; record data, observations, and any deviations from the prescribed procedure as you work; use blue or black ink. You must have the "landscape pages" with your data and observations signed by your TA before you leave the lab, and you will submit them with your lab report. Be sure to always record the units and the correct number of significant figures for any measured value.

Experiment 5:
Acid–Base Titrations

Pre-lab Questions (10pts)

*Pre-lab questions must be completed and turned in at the beginning of the lab period. If you have not completed the pre-lab questions, you will **not** be allowed to complete the lab.*

1. When the hydrochloric acid is added to the calcium carbonate, will there be an excess of HCl(aq) or $CaCO_3$(aq) or will there be stoichiometric equivalents of the two? Show your calculations. (4pts)

2. In this lab, you will use a 50mL buret and ~0.3M NaOH (aq). Calculate the mass of potassium hydrogen phthalate, KHP, needed for the standardization of NaOH (aq) in Part B. Show your calculations. Copy this number into the Procedure Section B. (3 pts)

3. Which indicator would you choose to perform titration in lab and why? What color change do you expect to see? (3pts)

Experiment 5

Name_____ **Partner(s)**_____

Procedure

SAFETY: **Safety goggles are required in lab.** HCl (hydrochloric acid) is corrosive, can cause chemical burns. NaOH (sodium hydroxide) is caustic and can cause chemical burns. Indicator solutions are potentially flammable and toxic; keep them away from open flames and do not ingest them.

Note – Ensure tip of burette is not chipped or broken and that valve turns easily and smoothly.

A. **Preparation of Acidified Antacid Tablet Solution**

1. Obtain two Tums antacid tablets to prepare solutions for two individual trials.
2. Weigh each antacid tablet on a weighing paper on the analytical balance to the nearest milligram. Record the mass.
3. Transfer the tablets to two clean 250mL Erlenmeyer flasks; one tablet in each flask.
4. Obtain HCl solution of approximately 0.300M concentration and recording the precise concentration. This value should be written on the label of the bottle.
5. Using a graduated cylinder, add 50.0mL of the HCl(aq) to each of the flasks.
6. Heat the flask to boiling on a hot plate. Gently boil the solution for about 5 min. While heating, use a glass stirring rod, if necessary, to help break up and disperse the tablets being careful keep all of the solid in its flask.
7. Set the solutions aside to allow them to cool to room temperature for the back-titration in Part C.

Data and Observations

Why are you boiling the solution in Step 6?

	Titration 1	Titration 2
Mass of tablet		
Concentration of HCl(aq)		
Volume of HCl(aq) added		

How could you improve the precision of the experiment?

B. **Standardization of NaOH Solution**

1. Accurately and precisely weigh out two (2) samples an appropriate amount of KHP (determined in pre-lab question 2) and record the values of each.
2. Dissolve each KHP sample in about 50 mL of water. This process may take a few minutes. Cover two of the solutions with paraffin film to stop them from reacting with the air and set them aside until you are to titrate them.
3. Add 2-3 drops of phenolphthalein indicator.
4. Begin adding the sodium hydroxide solution from the buret while continuously swirling the flask contents. A pink color will appear at the point where the NaOH mixes with the flask contents and will quickly disappear with swirling.
5. As the endpoint nears, this color will persist longer. At this point, do not open the stopcock completely.
6. Placing a white piece of paper under the flask will aid in observing the color change. When the color persists for 30 seconds after swirling, the endpoint has been reached.
7. Record the final buret reading to the appropriate number of significant figures.
8. Calculate the concentration in molarity of the NaOH solution (see calculations section).
9. Refill the buret so that you do not run out of NaOH solution in the middle of the next titration and repeat steps 3-8, two more times.
10. If the two determinations of concentration differ by more than 0.002 M, make a fourth measurement.
11. Use an average of these molarities for analyzing the antacid in the next part of the experiment. Your base solution is now standardized!

Why do you need to **accurately** measure the mass of KHP that you **estimated** in the pre-lab assignment?

Approximate mass of KHP need for standardization (answer from pre-lab, Question 2)

	Trial 1	Trial 2	Trial 3 (if needed)
Mass of KHP			
Initial buret reading			
Final buret reading			
Concentraion of NaOH solution (calculated)			

If your buret runs too low on NaOH, how can you continue the trial without having to start all over?

C. **Back-titration of the Antacid Tablets**

1. Refill the 50mL buret with the now standardized NaOH and record the initial buret reading.
2. Add 2-3 drops of the indicator you have been assigned to use to the acidified and cooled antacid tablet solutions.
3. Titrate by adding the sodium hydroxide solution from the buret while continuously swirling the flask contents.
4. Look for signs of an indicator change associated with your indicator. As the endpoint nears, this color will persist longer. At this point, you need to add NaOH solution more slowly.
5. When the color persists for 30 seconds after swirling, the endpoint has been reached. Record the final buret reading to the appropriate number of significant figures.
6. Repeat steps 1-5 for the second sample.

	Titration 1	Titration 2
Initial buret reading		
Final buret reading		

Also,

- Record the mass of active ingredient per tablet from the manufacturer's label.

Manufacturer's data	
Mass of active ingredient, $CaCO_3$	

Calculations

B. Standardization of NaOH solution

- Determine the number of moles of KHP used in each trial
- Determine the number of moles of NaOH added in each calibration titration.
- Determine the volume of NaOH solution added in each calibration titration.
- Determine the concentration of the NaOH solution for each trial.
- Find the average of value of the NaOH solution.

Include a sample calculation for each step. Then complete the table.

	Trial 1	Trial 2	Trial 3 (if needed)
Moles of KHP			
Moles of NaOH			
Volume of NaOH solution			
Concentration of NaOH solution			
Average of the concentrations			

Experiment 5

Name_____ **Partner(s)**_____

A and C. Back-titration of the Antacid Tablets

- Calculate the millimoles of HCl added.
- Calculate the millimoles of NaOH added.
- Calculate the millimoles of HCl that were neutralized by the antacid.
- Calculate the millimoles of $CaCO_3$ neutralized.
- Calculate the milligrams of the active ingredient, $CaCO_3$, in the tablet.
- Determine the percent mass of active ingredient per tablet from you experimental data.
- Determine the average percent mass from you data.

Include a sample calculation for each step. Then complete the table.

Experimental results	Titration 1	Titration 2
Millimoles of HCl initial (from Procedure Part A)		
Millimoles of NaOH titrant		
Millimoles of HCl neutralized		
Millimoles of $CaCO_3$ neutralized		
Mass of $CaCO_3$ neutralized		
Mass of tablet (from Procedure Part A)		
Percent mass of active ingredient, $CaCO_3$		
Average percent mass		

- Record the mass of active ingredient per tablet from the manufacturer's label.
- Record the mass of each tablet from the manufacturer's label.
- Determine the percent difference between your experimental value and the manufacturer's data.

Percent difference calculation example:

$$\frac{\%\ mass_{experimental} - \%\ mass_{manufacturer}}{\%\ mass_{manufacturer}} \times 100\%$$

Manufacturer's data	
Mass of active ingredient, $CaCO_3$	
Mass of tablet (average of your two measurements from Procedure Part A)	
Percent mass of active ingredient	
Percent difference experimental vs reported	

Experiment 5: Acid-Base Titrations

Post-Lab Questions (10pts)

1) If you follow the directions on the manufacturer's label about the number of tablets to be taken at one time, how many millimoles of HCl will be neutralized? Is this greater than the suggested maximum of 10 mmol HCl? (4pts)

2) A Rolaids tablet weighs about 1.4 g. According to the manufacturer it neutralizes 47 times its own weight of stomach acid. Each tablet contains 0.334g of the active ingredient, $NaAl(OH)_2CO_3$. Assume that the active ingredient reacts with HCl according to the following reaction:

$$NaAl(OH)_2CO_3\,(aq) + 4HCl\,(aq) \rightarrow NaCl\,(aq) + AlCl_3\,(aq) + 3H_2O\,(l) + CO_2\,(g)$$

a. How many moles of HCl will one Rolaids neutralize? (3pts)

b. Stomach acid is about 0.14M HCl and has a density about the same as water. Does Rolaids tablet neutralize 47 times its own weight of HCl or 47 times its own weight of 0.14M HCl solution (HCl + water)? (3pts)

Refrences:
Batson, W.B., and Laswick, P.H., "Pepsin and Antacid Therapy: A Dilemma," J. Chem. Educ. 56, 484-486 (1979).
Hem, S.L., "Physicochemical Properties of Antacids," J. Chem. Educ. 52, 383-385 (1975). Adapted from: Roberts, K.L, Hollenberg, J.L., Postmas, J.M., "General Chemistry in the Laboratory," 3rd Ed., W.H. Freeman and Co., New York, 1991