

**Volumetric Analysis & Consumer Chemistry**  
**Standardization of an unknown solution, analysis of vinegar & antacid tablets**

*OBJECTIVE:* The goals of this experiment are to learn titration concepts and techniques: to prepare and standardize a base solution and to use the standardized base to analyze vinegar & commercial antacid tablets. Using cost information, you will identify the brand of antacid that is a better buy.

*SKILLS:* Titration, manipulations of stoichiometric relationships involving concentration, back titrations

*EQUIPMENT:* Buret, Erlenmeyer flasks, hot plate

*REFERENCE Chemistry: An Atoms-Focused Approach*, Gilbert et al., 3<sup>rd</sup> edition, Sections 8.1, 8.2, 8.4, 8.5

*SAFETY AND DISPOSAL:* Water samples and glassware used in these exercises present no special hazards, although reasonable care should be used with NaOH solutions since they may damage clothing. Disposal of the liquid in the lab sinks and flushing with water is appropriate.

*INTRODUCTION:* Titration is a technique in which a measured volume of a solution of one reactant is added to a second reactant whose quantity is to be determined. When the number of moles of the first reactant is equivalent to the number of moles of the second, a change, usually in color, occurs in the system. This signals the titration endpoint and permits determination of the unknown quantity. In this experiment, various acidic solutions will be titrated with a solution of NaOH using an indicator that turns pink when the endpoint is reached.

In this experiment, the titration process involves preparing (making up) and standardizing (accurately determining the concentration of) a basic solution of NaOH using a known quantity of a chemically pure and dry standard acid, potassium hydrogen phthalate, ( $\text{KHC}_8\text{H}_4\text{O}_4$  MW =204.2g), often abbreviated as KHP. In order to use your NaOH solution for further analysis, you must perform several trials of your standardization.

You will perform two analyses with your standardized NaOH solution. First, you will determine the acid content of commercial vinegar. Vinegar is a solution of acetic acid ( $\text{CH}_3\text{COOH}$  or  $\text{HC}_2\text{H}_3\text{O}_2$ ) in water. In addition to the acetic acid, there are usually small amounts of trace substances which give vinegar a subtle flavor and complement its acid (sour) taste. Different vinegars may have different acetic acid concentrations and different trace substances. Most commercial white vinegars have an acetic acid concentration of less than 5 % by weight.

You will also determine the amount of base in a commercial antacid tablet. High acidity is used for digestion of food in human stomachs. If there is too much acidity, however, an individual may feel sick. To counteract this, antacid tablets (containing basic chemicals that can neutralize some of the acidity) are often taken. Different tablets may have different strengths as will be shown in this experiment. In the analysis of the antacid tablets, a back titration is performed. In this case, an excess amount of standardized acid is allowed to react with the antacid tablet. When the reaction is complete, the amount of acid unreacted (or left over) is determined via a titration with your standard NaOH. The difference between the known amount of acid that was originally placed in the flask and the amount of acid that reacts with the NaOH is the amount of acid that reacted with the antacid.

#### EXPERIMENTAL PROCEDURE

**BE SURE TO WEIGH YOUR CRUCIBLE AND PRODUCT FROM EXPERIMENT 6 THAT HAS DRIED OVER THE WEEK IN YOUR LOCKER. RECORD THE MASS AND ENTER THE DATA ON THE LAB COMPUTER.**

**You will perform this experiment over two weeks to allow for development of your titration skills. The NaOH you standardize during the first week will be stored in plastic bottles to use for the analysis of vinegar and antacid during your next lab meeting. You must complete the standardization during week one.**

#### **Preparation of the NaOH solution**

Prepare 500 mL of approximately 0.1 M NaOH solution by calculating the volume of 1 M NaOH needed for dilution with water to a total volume of 500 mL. Take that amount of 1 M NaOH, place it in a 600 mL beaker, add water until the total volume is about 500 mL, and stir. In the experiment that follows, this solution will be standardized and then used for the titration. Transfer your prepared solution to a labeled plastic bottle. The bottle should be labeled with the following information:

*Your Name*  
CHEM 2115 – Section *XXX*  
*XXXX* M Sodium Hydroxide  
CAS# 1310-73-2  
CORROSIVE

You will replace the “XXXX” with the actual concentration after you perform the standardization. Note that if the solution is not properly prepared, stored, or if it is all used up a new solution must be made and standardized.

(Caution: Be careful to work neatly and to wipe up any spills. Note that if drops of NaOH solution are left on the surface of a lab bench by any lab worker, damage to skin, books, clothing, or other valuables may result.)

## I. Standardization of the NaOH solution

The process that follows will accurately determine the concentration of the standardized NaOH solution. Prepare a buret by rinsing it with two 10 mL portions of the NaOH solution. Then fill the buret with the NaOH solution. Record the starting reading of the buret. **Be sure that there are no air bubbles in the buret and that the tip of the buret is filled.** This solution will be used for the titrations described below.

Into each of your clean 250 mL Erlenmeyer flasks, weigh between 0.280–0.320 g of  $\text{KHC}_8\text{H}_4\text{O}_4$  (potassium hydrogen phthalate, commonly abbreviated as KHP, with  $\text{MW} = 204.2 \text{ g/mol}$ ). Be sure to record the actual mass added to each flask. The transfer of the solid KHP to your flask should be done quantitatively by rinsing the weigh boat with distilled water into your flask several times. Add about 75 mL of distilled water to each flask and swirl it to dissolve the solid. After all of the KHP has dissolved, add 3 drops of phenolphthalein. **All of the KHP must be dissolved before you begin titrating.**

### *Rough Titration*

It is useful to perform a rough titration to get an idea of about how much of the titrant (your NaOH solution) will be needed to reach the end point. In subsequent trials you can then add titrant from the buret quickly at first and then slowly as you get near the end point. Use your sample with the **smallest** mass of KHP for your rough titration. Record the starting reading of the buret. Next, add NaOH from your buret in about 1 mL increments. After each addition, swirl your flask and observe the color of the solution. Continue adding NaOH in 1 mL increments until you achieve a pink color that remains for 30 seconds. **NOTE: You will most likely get a very dark pink color. This is acceptable for the rough titration ONLY since the dark color is an indication you have surpassed the end point. A rough titration is NOT included in your calculations since you surpassed the endpoint.**

Record the final reading of the buret and determine the volume of titrant added. This will give you a rough idea of the amount of base needed in your subsequent standardization trials. For example, if your rough titration required 10 mL of base, then in your quantitative trials you can add about 8–9 mL of NaOH from the buret quickly. Then slowly add NaOH drop by drop to reach the end point, as described below.

### *Quantitative Trials*

Now that you have a rough idea of the amount of base needed to reach the end point you will be perform a minimum of three quantitative trials. In these trials it is essential you reach a quality endpoint, which is evidence by a **light** pink color that remains for 30 seconds. A bright pink color similar to your rough titration indicates you have surpassed the end point and that trial is not valid.

Refill your buret before beginning your next titration. To find the amount of NaOH used, the buret must be read carefully before and after the titration begins. Before titrating, record the initial reading on the buret. **Volumes in burets should be read to two decimal places.** When the titration is complete, again record the volume reading on the buret. The volume added during the titration can be found by difference.

Begin your quantitative trials by adding a volume of NaOH as determined by your rough titration. As described above, use 1–2 mL less of NaOH than used for your rough titration. This will help avoid surpassing the endpoint during this addition.

Next, begin to slowly add NaOH from the buret into the flask containing KHP. Be sure to swirl the solution carefully after each addition. As you get closer to the end point, the pink color will remain in the flask longer when you swirl. At this point you should begin to add NaOH drop by drop. Titrate until the **light** pink color of the phenolphthalein indicator **just** appears and then remains for 30 seconds. It is helpful to place a white piece of paper under your flask to help you see the light pink color.

The neutralization reaction taking place is  $\text{HKC}_8\text{H}_4\text{O}_4(\text{aq}) + \text{NaOH}(\text{aq}) \rightarrow \text{H}_2\text{O} + \text{Na}^+(\text{aq}) + \text{K}^+(\text{aq}) + \text{C}_8\text{H}_4\text{O}_4^{2-}(\text{aq})$

Determine the volume of NaOH used in the titration. Repeat the procedure to obtain a total of three quality trials. This information can be used to find the concentration of the NaOH solution, since at the end point the number of moles of NaOH added from the buret is equal to the number of moles of KHP you began with. Both the number of moles of KHP and the volume of NaOH used are known, so the concentration of the NaOH can be determined. Calculate the concentration of the NaOH solution in each of the three quantitative trials and average the results. **Show your results to your instructor to see if you can proceed to the next analysis.** Enter the concentration of your NaOH solution data into the lab computer.

***Notebook template information for Part I.*** In addition to your observations, be careful to record the mass of KHP used and the initial and final volumes of NaOH in your buret. From the mass of KHP, you can determine the moles of KHP. From the buret volumes of NaOH, you can find the volume of NaOH solution used. From this information you can determine the molarity of your NaOH solution for each trial and you can find the average NaOH molarity.

## II. Vinegar Analysis

You will weigh the vinegar used in each trial by difference. Measure a 2.0 mL sample of vinegar into a 10 mL graduated cylinder. Determine the mass of the cylinder with the vinegar. Transfer the vinegar to a clean 250 mL Erlenmeyer flask and reweigh the 10 mL graduated cylinder. The difference in mass is an accurate measure of the mass of vinegar transferred. Dilute the sample with about 15 mL of distilled water and add 3-4 drops of phenolphthalein. Titrate the vinegar using the NaOH solution that you standardized in Part I. You may wish to perform a rough titration as described in Part I.

For three quantitative trials (light pink endpoint), record the volume of NaOH used. Calculate the number of moles of NaOH and acetic acid that reacted, and determine the number of grams of acetic acid that were in the vinegar sample. Use this information to calculate the weight percent of acetic acid in vinegar. In the lab report, you will calculate the weight percent of acetic acid in your vinegar sample by averaging the results of the three determinations.

*Notebook template information for Part II.* For each trial, record the mass of your graduated cylinder with vinegar and of the graduated cylinder after the vinegar has been transferred from it. Determine the mass of vinegar used. Record the initial and final volumes of NaOH used and determine the volume of NaOH used. Find the number of moles of NaOH and acetic acid, and use the result to determine the weight percent of acetic acid in vinegar for each trial. Find the average value for the weight percent of acetic acid in vinegar.

## III. Antacid Analysis

Accurately transfer 0.340–0.360 g of a pulverized commercial antacid to a 250 mL Erlenmeyer flask. Add about 20 mL of distilled water to the flask and then pipet 5.00mL of standardized HCl to the flask using a transfer pipet and swirl the mixture to dissolve the antacid. Be sure to record the concentration of the standardized HCl. Some solid may remain and is usually unreacted tablet “fillers”. You will be performing several trials of the antacid analysis so you will need several samples of the same antacid.

Slowly heat the solution to boiling and continue to heat at a gentle boil for at least 1 minute to expel the dissolved CO<sub>2</sub>. Add 4-8 drops of phenolphthalein indicator to the flask. If the solution is pink, add an additional 5.0 mL of standardized HCl to the flask and boil again for 1 minute.

Titrate the excess standard HCl to the endpoint using the standardized NaOH you prepared in part I. It would be useful to perform a rough titration as you did previously. Record the initial and final volumes of NaOH in the buret and determine the total volume of NaOH used. Perform a total of **three** quantitative trials using the same antacid. Enter your data into the lab computer.

**Notebook template information for Part II.** For each trial, record the mass of antacid used. Record the molarity and volume of standardized HCl used. Record the initial and final volumes of NaOH used and determine the volume of NaOH used. Find the number of moles of NaOH used in the titration, which is equal to the number of moles of excess hydrochloric acid, i.e. the moles leftover after some of the HCl has been neutralized by the antacid. Determine the moles of HCl that were neutralized by the antacid by difference. This value is equivalent to the number of moles of base that the antacid contained, because this was the number of moles of base that would have neutralized the same amount of acid. Finally, because a large sample of antacid would neutralize more than a small sample, it is useful to find the number of equivalent moles of NaOH per gram of antacid.

## LAB REPORT

The Week 1 lab report for this experiment will include the determination of the solution molarity from Part I.

The Week 2 report will include a determination of the weight percent acetic acid in vinegar from Part II and the number of equivalent moles of NaOH per gram of antacid from Part III. Data will also be available for the cost per gram of the antacid tablets. As part of the comparison of different commercial antacids, you will determine the cost per equivalent mole of base in the antacid. That is, you will find how much it costs when different antacids neutralize the equivalent amount of HCl as one mole of base would. The Week 2 will also include an analysis of the class data for the antacid titrations.

Class data for the antacid analysis can be downloaded from a Blackboard file in which each student will have entered the molarity of their NaOH solution and data on their antacid trials. Tests were done on two different brands of antacids. In the class data part of the report, you will use Excel to find the equivalent moles of base per gram of tablet for each measurement made in the class, and then determine the average values for both types of antacid samples. The calculations for the analysis of class data involve new Excel techniques, described in the section below.

### *Excel Instructions for Class Data Calculations*

There are many ways to use Excel to calculate the required values. One way is to first separate the data into groups reflecting the brand of antacid. The sorting of the data was performed before the data was uploaded to Blackboard.

*Antacid calculations:* The ultimate goal is to calculate the cost of the antacid per mole of base it provides. However, the first step is to determine the moles of base provided by the antacid per gram of antacid:

Equivalent moles of antacid (base) / g antacid = (initial moles HCl added – mol HCl leftover (titrated) ) / g antacid

Next, the cost per gram of antacid can be used to determine the cost per mole of base in the antacid. This number can then be compared for the two brands to determine which is a better buy. The following sequence of steps (each step has a new column in the Excel data file) can be used to find the cost per mole of base:

1. Find the initial number of moles of HCl added by multiplying the molarity of the HCl solution (cell H3) times the volume of HCl added initially (column C) in liters. For example, if you are working on the data in Row 11 (the first row with data) you can enter the following formula in cell D11:  $=\$H\$3 * (C11/1000)$ . The dollar signs (\$) create a fixed cell reference (the molarity of the HCl is the same for each sample). You can then copy and paste this formula into the remaining cells in column H.
2. Next find the number of moles of base you added in the titration with the leftover HCl. To do this, multiply the molarity of the NaOH (column F) times the volume of NaOH titrated in liters. The formula for row 11 in column G would read:  $=F11 * (E11/1000)$ . The NaOH you added from the buret during the titration reacted with the leftover HCl that did not react with the antacid. For each mole of NaOH added, one mole of leftover HCl is neutralized. Thus, the number of moles of NaOH added during the titration equals the number of moles of HCl leftover.
3. Now it is possible to find out how many moles of HCl reacted with the antacid:  
 $\text{moles HCl reacted with antacid (H)} = \text{initial moles HCl added (D)} - \text{leftover moles of HCl (G)}$   
 where the bold letter indicates the corresponding column in the Excel worksheet. For the data in row 11, the formula in cell **H11** would read:  $= D11 - G11$ .
4. For every mole of HCl reacted, one mole of antacid (base) also reacted. Therefore, the moles of base provided by the antacid (column I) equals the moles of HCl reacted (column H).
5. We're now interested in how much base is in the antacid per gram of tablet. This will enable us to compare the two brands. The mass of each antacid sample is recorded in column J. Therefore, to find the moles of base in the antacid per gram of tablet, divide the value in column I by the value in column J. This can be done in column K. For example, in K11 you might write:  $= I11 / J11$ .
6. Finally, we want to find out which brand is a better buy. To do this we must make use of the data for the cost of the antacid per gram. If we take the cost per gram of tablet and divide by the moles of base in the antacid per gram of tablet, we get the cost per mole of base in the antacid (a value we can use to compare the two brands):

$$\frac{\frac{\text{cost (dollars \$)}}{\text{gram antacid}}}{\frac{\text{moles base}}{\text{gram antacid}}} = \frac{\text{cost (dollars \$)}}{\text{gram antacid}} \times \frac{\text{gram antacid}}{\text{moles base}} = \frac{\text{cost (dollars \$)}}{\text{moles base}}$$

The cost per gram can be found in cells **H4** for Roloids and **K4** for Tums. So, for example if row 11 was a Roloids sample, then in cell L11 you would write:  $= \$H\$4 / K11$ .

Look at the antacid data; are any of the values obviously wrong? If so, make a note of which ones and set those values to a blank. Once this is finished, the results can be averaged in the same way that we have done previously.

In doing complicated calculations like these, it is often useful to do one calculation by hand so that you can compare it to the Excel result for a particular case. You will have already done this in your answer Part III, so you can check to see if you told Excel how to do it correctly.

*Relative error analysis:* Compare the results for your antacid samples to the class values by calculating their relative errors. The relative error is defined as the error (the difference between your value and the average value) divided by the average value. **It is often given as a % error.** A positive (negative) relative error means that your value was higher (lower) than the class average.

$$\text{Relative Error} = \frac{\text{Your Value} - \text{Average Value}}{\text{Average Value}} \times 100$$