Ascorbic Acid Titration of Vitamin C Tablets

This lab will be completed **individually**! Make sure you come prepared!

Introduction

Vitamin C (also known as ascorbic acid, $HC_6H_7O_6$) is a necessary ingredient in the human diet. A deficiency of Vitamin C leads to the disease scurvy, at one time commonly occurring during long sea voyages. British sailors combatted scurvy by carrying limes, rich in Vitamin C, on their voyages, thus engendering the name "limey." Although the Food and Drug Administration recommends a daily intake of 60 mg of Vitamin C, Linus Pauling suggested that amounts of 1-2 grams daily are instrumental in fighting the common cold.

This experiment illustrates how titration can be used to determine the ascorbic acid content of a Vitamin C tablet containing about 500 mg of Vitamin C. First, you will determine the concentration of a sodium hydroxide solution using a standardized solution of sulfuric acid. The mass percentage of ascorbic acid in Vitamin C will then be determined by titrating the Vitamin C samples with the standardized sodium hydroxide solution.

In some acid-base neutralization reactions, an acid reacts with a metal hydroxide base to produce water and a salt:

$$\underset{\text{Acid}}{HX\ (aq)} + \underset{\text{Metal hydroxide base}}{MOH\ (aq)} \rightarrow \underset{\text{water}}{H_2O\ (l)} + \underset{\text{salt}}{MX\ (aq)}$$

The ***protons*** (H^+) from the acid react with the ***hydroxide ions*** (OH^-) from the base to form the water. The salt forms by combining the cation from the base and the anion from the acid. Acids often react with bases; the solubility of the salt does not determine whether the reaction occurs or not. The solubility of the salt and its state can be determined by reading the solubility rules.

In this experiment, you will determine the molarity (or molar concentration) of NaOH (aq) using a standardized H_2SO_4 (aq) solution. A ***standard solution*** has been analyzed, so its concentration is known to a certain degree of accuracy. The H_2SO_4 (aq) solution used in this laboratory was standardized in our stockroom to four significant digits. Write the balanced equation for the reaction that occurs between sodium hydroxide and sulfuric acid on your report sheet.

You will measure out a small volume of sulfuric acid and use a ***buret*** to determine the volume of sodium hydroxide required to completely neutralize the acid. The process of slowly adding one solution to another until the reaction between the two is complete is called a ***titration***.

When carrying out an acid-base neutralization reaction in the laboratory, you observe that most acid solutions and base solutions are colorless, and the resulting water and soluble salt solutions are also colorless. Thus, it is impossible to determine when a reaction has occurred, let alone when it is complete.

To monitor the progress of a neutralization reaction, you will use an ***acid-base indicator***, a solution that changes color depending on the pH (or acid-content) of the solution. One commonly used indicator is ***phenolphthalein***, which is colorless in acidic and neutral solutions and pink in basic (or alkaline) solution. During a titration, the indicator is added to the sample being analyzed. The ***titrant*** is slowly added to the sample until the ***endpoint*** (when the indicator changes color) is reached, signaling that the reaction between the two is complete. Note that phenolphthalein turns pink only when **excess** sodium hydroxide has been added.

If the appropriate indicator has been chosen, the endpoint of the titration (i.e., the color change) will occur when the reaction is complete, or when the acid and base are stoichiometrically equivalent:

moles of acid = moles of base

A Vitamin C tablet contains ascorbic acid, $HC_6H_7O_6$ (aq), as well as binder material that holds the tablet together. The balanced equation for the reaction between ascorbic acid and sodium hydroxide is shown below:

$$HC_6H_7O_6\ (aq) + NaOH\ (aq) \rightarrow H_2O\ (l) + NaC_6H_7O_6\ (aq)$$

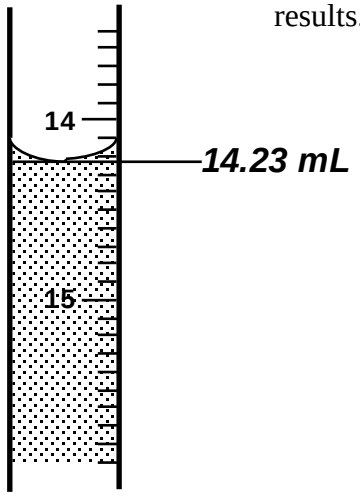
You will titrate each Vitamin C sample with the standardized NaOH solution to determine the mg of ascorbic acid present in each sample.

Techniques:

1) Burets

Burets are used when it is necessary to deliver a liquid to another container and record the exact amount delivered. A buret is marked in milliliters much like a graduated cylinder, except buret markings indicate the number of milliliters **delivered**. This means that **0** (none delivered) is at the top, and the numbers get larger as you go down the buret. The stopcock controls the liquid flow. It is open when parallel to the length of the buret and closed when parallel to the floor.

- **Rinsing and conditioning the buret:** Obtain some deionized water in a small beaker. With the buret over the sink and the stopcock open, pour the water through the buret, letting it drain out the tip into the sink. After the buret is well-drained, close the stopcock and use a small beaker to pour 5-10 mL of the solution to be used (NaOH for this lab) into the buret. Tip the buret sideways and rotate to completely rinse the inside of the buret. Run this solution through the buret tip into your 400 mL waste beaker. This will prevent dilution or contamination and give more accurate results.
- **Filling the buret:** Close the stopcock. Use the beaker of NaOH and a funnel to fill the buret 1 mL above the "0" mark. Place a container under the buret tip and open the stopcock slowly. The buret tip should fill with solution, leaving no air bubbles. If the tip does not fill with solution, ask the instructor for help. Continue to let out solution until the liquid level is at "0" or below.
- **Reading the buret:** Record the volume by noting the bottom of the meniscus. (Be sure that the meniscus is at eye level). If this reading is exactly "0," record 0.00 mL. Otherwise, count the number of markings between each number, and estimate to the nearest 0.01 mL.



Thus, in the example on the previous page, the meniscus is about one-third of the way between 14.2 and 14.3, so the volume is recorded as ***14.23 mL***.

Buret readings are always recorded in mL to <u>2 decimal places</u>.

- Deliver the required volume (usually when you get a color change). To calculate the volume of solution delivered, subtract the initial volume from the final volume.
- ***Always refill your buret before each trial***, so you do not need to refill the buret during a trial. Refilling the buret in the middle of a trial reduces accuracy. When you are finished, empty the buret, and rinse it with DI water, allowing some water to run through the tip.

2) Pipets

You will use a pipet to deliver 10.00 mL of H_2SO_4 to an Erlenmeyer flask. For best results, make sure that the top of the pipet and the bottom of the pipet bulb are dry before use (Note: While a pipet bulb is mentioned in the following text, there are other devices that may be used to draw in solutions to the pipet). You will first condition the pipet (similar to conditioning a buret for a titration). Place the pipet bulb loosely on the top of the pipet, squeeze the bulb, position the tip of the bulb below the liquid level (in your beaker), and slowly release the bulb to draw up a **small amount** of liquid into the pipet, making sure that the tip of the pipet stays below the liquid level. Remove the bulb and quickly slide your index finger or thumb over the top of the pipet. Holding the pipet almost horizontally over a waste beaker in the sink, rotate the pipet in order to coat the inside of the pipet with the solution it will contain. Allow the solution to drain out into your 400 mL waste beaker.

- Replace the bulb, squeeze it, and position the pipet as before. This time, fill the pipet well above the calibration line (etched or marked above the wide center section of the pipet), **taking care not to get liquid into the pipet bulb**. (If this happens, let your instructor know.)
- Slide the bulb off the pipet while quickly sliding your index finger or thumb over the top of the pipet. Move your finger slightly and rotate the pipet to allow the liquid level to drop to the calibration line on the pipet. Then press down harder with your finger and transfer the tip of the pipet into a position over the Erlenmeyer flask.
- Remove your finger and allow most of the liquid to drain out. Then hold the tip of the pipet against the inside of the flask for about 10 seconds to allow more liquid to drain.
- **Do not** try to remove the small amount of liquid remaining in the tip. Pipets are calibrated to retain this amount.

Example Calculations

Titrating an Acid:

Consider the following reaction between hydrochloric acid, HCl(aq), and sodium hydroxide, NaOH (aq),

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

In a titration scenario, the concentration of HCl(aq) is unknown. Using a standardized sodium hydroxide solution with a concentration of 1.020 M, a student titrated 25.00 mL of hydrochloric acid. If 27.14 mL of sodium hydroxide was required to completely neutralize the hydrochloric acid to a faint pink phenolphthalein endpoint, the molarity of the hydrochloric acid is calculated as follows.

The first step in this calculation is recognizing that you are solving for the molarity of hydrochloric acid, which has units of moles per liter and which we can represent as [HCl] (Note: chemists sometimes denote concentration by using brackets as short-hand. For example, "[HCl]" would mean "concentration of HCl"):

$$\text{molarity of HCl} = [\text{HCl}] = \frac{\text{mol HCl}}{\text{L HCl}}$$

Since 25.00 mL of hydrochloric acid was used, convert that to liters (by dividing by 1000, which moves the decimal point to the left three places), and put it in the denominator:

$$\text{molarity of HCl} = [\text{HCl}] = \frac{\text{mol HCl}}{0.02500\ \text{L HCl}}$$

To determine the number of moles of hydrochloric acid, the number of moles of NaOH must first be calculated. ❶ Convert the volume of sodium hydroxide from milliliters to liters then ❷ multiply that by the molarity of sodium hydroxide (given as 1.020 M and shown below as a conversion factor).

By showing the molarity as a fraction (M = mol/L), you can see that the volume units (liters of NaOH) cancel. Now the moles of hydrochloric acid can be calculated in the next step ❸ by using the mole-to-mole ratio between sodium hydroxide and hydrochloric acid found in the balanced chemical equation. The complete calculation to get moles of hydrochloric acid is shown below:

$$\overset{\text{❶}}{} \qquad \overset{\text{❷}}{} \qquad \overset{\text{❸}}{}$$

$$27.14\ \text{mL NaOH}\left(\frac{1\ \text{L}}{1000\ \text{mL}}\right)\left(\frac{1.020\ \text{mol NaOH}}{\text{L of NaOH}}\right)\left(\frac{1\ \text{mol HCl}}{1\ \text{mol NaOH}}\right) = \mathbf{0.02768\ mol\ HCl}$$

Notice that in the equation above, we know where to place each number (numerator or denominator) by checking if the units cancel.

Finally, we put the number of moles of HCl in the numerator or concentration expression, and the molarity for hydrochloric acid is calculated as follows:

$$\text{molarity of HCl} = [\text{HCl}] = \frac{0.02768\ \text{mol HCl}}{0.02500\ \text{L HCl}} = \mathbf{1.107M\ HCl}$$

Note that there are ***4 significant figures*** in all calculations for this experiment.

Ascorbic Acid Titration of Vitamin C Tablets

Part A. Preparation of Vitamin C Tablet Solutions

1. Obtain two vitamin C tablets. Place a plastic weighing boat on the balance, and press zero to tare the balance. Place the pieces in the tared weighing boat, and record the exact mass of the tablet in your **LAB NOTEBOOK**. MAKE SURE TO RECORD **ALL** OF THE DIGITS! Repeat this procedure for the second tablet in a separate plastic weighing boat.

2. Crush each tablet by placing it between two plastic weighing boats and applying firm pressure with a pestle, as demonstrated by your instructor. Transfer the tablets to separate, labeled 250 mL Erlenmeyer flasks. Add 40-50 mL of deionized water to each sample. Use a hotplate at your lab bench to heat the flasks (heat setting between 3-4). Heat gently for about ten minutes to dissolve the vitamin C tablets. The binder in the tablet will not completely dissolve, leaving some residue. Set these solutions to the side to cool to room temperature while you complete the titrations in Part B.

CAUTION: Sodium hydroxide, NaOH, can cause chemical burns and damage eyes very quickly. Any NaOH spilled on your skin must be rinsed immediately with water for 15 minutes. Any NaOH spilled on the lab benches should be neutralized, and the area rinsed with water and wiped clean. Inform your instructor of any NaOH spills.

CAUTION: Sulfuric acid, $H_2SO_4(aq)$, is corrosive and can cause chemical burns and damage clothing. Any $H_2SO_4(aq)$ spilled on skin must be rinsed immediately with water for 15 minutes. Any acid spilled on your work area must be neutralized, the area rinsed with water and wiped clean.

WEAR GOGGLES AT ALL TIMES, even when you are washing the glassware to avoid exposing your eyes to NaOH solution. Wash your hands completely with soap and water before leaving the lab.

Part B. Standardization of the NaOH Solution

LAB NOTEBOOK

You will need to make a data table which contains the following information:

- Initial, final, and total volumes (in mL) for *each* titration trial.
- Calculated molarity of NaOH for each trial

You may want to save room under your table to show calculations

1. Prepare a data table in your lab notebook to record volume and concentration data as suggested above.

2. Use the NaOH pump dispenser to deliver 100 mL of NaOH into a clean, labeled 250 mL beaker. Clean, rinse, and condition a 25.00 mL buret with a few mL of the NaOH solution, then fill the buret with the NaOH solution. Drain a small amount of the NaOH solution into your waste beaker so it fills the buret tip (with no air bubbles present). Record the exact initial buret reading. (Save the rest of the NaOH solution in the beaker to refill the buret later.)

3. Use the H_2SO_4 pump dispenser to dispense 40 mL of H_2SO_4 into a clean labeled 150 mL beaker. **Record the exact concentration of H_2SO_4 from the label on the dispenser in your LAB NOTEBOOK either directly above or below your data table**.

4. **Review how to properly use a pipet found in the Background Section before continuing.** Using the 10.00 mL pipet and a rubber bulb (or other device such as a pipet pump), pipet 10.00 mL of the standard H_2SO_4 solution into a *clean* 250 mL Erlenmeyer flask. Only gravity should be used to deliver the acid solution from the pipet to the flask. In other words, don't use the bulb or pump to force the solution out of the pipet. Also remember that the pipets are *designed* to leave a small amount of liquid in the tip (do not blow it out!). Add about 10 mL of deionized water and 2 drops of phenolphthalein indicator to the acid. Repeat for the other two flasks. The 10 mL of water that you add does not need to be exact. Do **NOT** use the pipet to measure the 10mL.

5. Place a teflon magnetic stir bar in the Erlenmeyer flask. Place the flask on a cool stir plate. Adjust the stir setting so that your solution is contiuously being mixed without splashing the solution on the insides of the flask.

6. Slowly add the NaOH from the buret to the acid solution in the flask, while swirling the flask to get homogeneous solutions. When you begin seeing flashes of pink, add the base dropwise, occasionally rinsing the sides of the flask with deionized water from a wash bottle. (*Note: The slower the NaOH is added near the end of the titration, the more accurately you can catch the endpoint. The closer you stop the titration at the endpoint, the less likely you will have to redo a trial.*) Stop adding base when one drop causes a permanent (>1 minute) **faint pink** coloration of the solution in the flask. Record the reading on the buret at this endpoint ***to the nearest 0.01 mL***.

7. Refill your buret with the NaOH solution. Titrate the other two H_2SO_4 samples with the NaOH solution. Record the exact initial and final buret readings in your data table. When titrations are performed, a minimum of three trials should be completed to ensure accuracy. More trials should be completed if any volume of NaOH used differs by more than 1 mL.

Part C. Analysis of Ascorbic Acid in Vitamin C Tablets

LAB NOTEBOOK

You will need to make a data table which contains the following information:

- Initial, final, and total volumes (in mL) for *each* titration trial.
- Calculated mass of ascorbic acid (in mg) from the titration data for each tablet

Add 2 drops of phenolphthalein solution to each flask containing a Vitamin C tablet. Titrate each sample (i.e., 2 trials) with the NaOH solution to pink phenolphthalein endpoints. For Part B, the pink color of the endpoint will persist for a long time. For the titration of the ascorbic acid in the tablets, the pink color will disappear fairly rapidly due to the slow reaction between the binder in the tablet and the sodum hydroxide. This reaction removes the base from the solution. Below the data table you will need to calculate the average mass of Ascorbic acid (in mg) and the average mass % of Ascorbic acid from both trials. In the interest of time, you will only titrate two samples of Vitamin C tablets. For accuracy, it would be ideal to perform 3 titrations in this step as well, but time constraints don't allow this.

Waste Disposal: Combine all solutions in your waste beaker and dispose in waste container in the hood.

BE SURE TO WASH AND DRY YOUR LAB BENCH AFTER COMPLETING THE EXPERIMENT TO REMOVE ALL TRACES OF ANY SPILLED CHEMICALS

Calculations to be Completed in LAB NOTEBOOK

Part B. Standardization of the NaOH Solution

1. Write the balanced chemical equation for the reaction between sodium hydroxide and sulfuric acid.

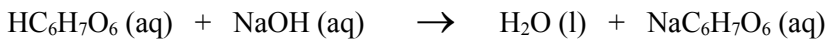
2. Calculate the molarity of the NaOH from the data for each titration of H_2SO_4(aq). Don't forget to take into account the mole to mole ratio of the two substances from the balanced chemical equation. Make sure to label each calculation such that it can be graded.

3. Calculate the average molarity of the NaOH(aq).

4. After completing the calculations, be sure to complete your data table.

Part C. Determining the Amount of Ascorbic Acid in Vitamin C

1. Use the average molarity of the NaOH from part B to calculate the moles of ascorbic acid present in your flasks. Then convert moles of ascorbic acid to grams and then milligrams. The equation for this reaction is shown here (as always, check to see if the equation is balanced):



$$HC_6H_7O_6\,(aq) + NaOH\,(aq) \rightarrow H_2O\,(l) + NaC_6H_7O_6\,(aq)$$

(Write this chemical reaction equation in your lab notebook)

2. Calculate the average mass of ascorbic acid in your two tablets.
3. Be sure to label each calculation, and complete your data table.

For Your Lab Report:

Attach the copies of your lab notebook and the Postlab Questions, pages 8-9, and submit as your report.

Ascorbic Acid in Vitamin C Tablets: Lab Report

Name: _____

Section Number:_____

Turn in pp. 8 and 9 with the copies from your lab notebook

Post-Lab Questions

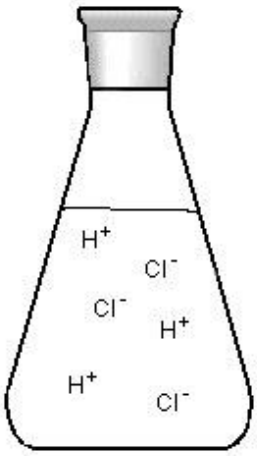
1. In acidic solutions, phenolphthalein is: (Circle one) pink colorless

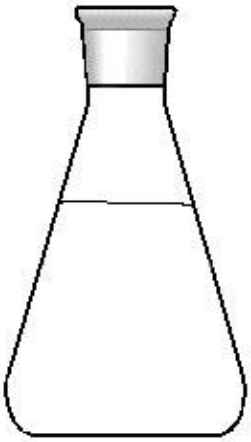
   In basic solutions, phenolphthalein is: (Circle one) pink colorless

2. Acidic solutions contain what ions, specifically? _____

   Basic solutions contain what ions, specifically? _____

3. Hydrochloric acid can also be titrated with sodium hydroxide using phenolphthalein indicator to determine the endpoint. The Erlenmeyer flask on the left below shows that the only ions present at the start of the titration are $H^+(aq)$ and $Cl^-(aq)$. Indicate the color of the solution at the start. For the second flask, write the chemical formulas for the substances present (other than water) at the endpoint of the titration between hydrochloric acid and sodium hydroxide. Also indicate (by circling) the color of the solution at the endpoint.





Before any NaOH(aq) is added, the solution is: pink colorless

At the endpoint of the titration, the solution is: pink colorless

4. In Procedure B, Step 2, why wasn't it necessary to record the exact volume of water added to the flask?

4. Explain, in terms of substances present, why the solution in the flask, after a few milliliters of NaOH(aq) have been added, turns pink for a few seconds then becomes clear again.

5. Explain, in terms of substances present, why the solution in the flask turns pink and stays pink at the endpoint.

6. How does the average milligrams of ascorbic acid that you calculated (refer to Part C calculations above) compare with the manufacturer's claim of 500 mg of ascorbic acid per tablet? Describe at least three sources of error <u>in your lab techniques</u> that could have resulted in different amounts of ascorbic acid than the manufacturer's claim.

7. How would the ***calculated*** **molarity for NaOH** be affected (higher, lower, or no change) if the following procedural errors occurred? Explain why in each case.

   a. While pipetting the H_2SO_4 solution, several drops of H_2SO_4 drip out of your pipet onto the bench top and miss the Erlenmeyer flask.
   - NaOH molarity (circle one): high or low
   - Why?

   b. The buret tip is not filled with NaOH at the beginning of the titration.
   - NaOH molarity (circle one) : high or low
   - Why?

9. Predict the products (including phases) and balance the equation for each of the following sets of reactants:

a.	HNO_3 (aq) + $Ba(OH)_2$ (aq) $\rightarrow$
b.	HBr (aq) + LiOH (aq) $\rightarrow$
c.	$HC_2H_3O_2$ (aq) + $Ca(OH)_2$ (aq) $\rightarrow$
d.	H_3PO_4 (aq) + KOH (aq) $\rightarrow$