Lab 10: Acid-Base Titration

Objectives: To learn the technique of titration and apply it to determine the concentration of acetic acid in vinegar by titration with sodium hydroxide.

Materials: 250 mL Erlenmeyer flask; 150 mL beaker; 50 mL buret; 5 mL pipet; pipettor; vinegar solution; stirrer; magnet bar; approximately 0.10 M NaOH solution (standardized sodium hydroxide); and phenolphthalein indicator, pH meter.

Safety: Sodium hydroxide solution is very caustic. It can cause skin burns and is extremely damaging if it gets in your eyes. Always wear gloves and safety goggles when working at the bench with sodium hydroxide.

Waste Disposal: All solutions must be discarded into the designated inorganic waste container.

INTRODUCTION

The concept of acid-base behavior is one of the most fundamental in chemistry, with important applications in biochemistry and industry. Arrhenius definition of an **acid** is a substance that acts as a proton (H^+) donor and, in aqueous solution, produces hydronium ions (H_3O^+) and a **base** acts as a proton acceptor and, in aqueous solution, produces hydroxide ions (OH^-).

The Brønsted-Lowry definition of an **acid** is a **proton donor** (where a proton is an H^+ ion) and a **base** is a **proton acceptor**. A substance that can act as either an acid or a base is termed **amphiprotic** or **amphoteric**. Acids and bases can be characterized as **strong** or **weak**, depending on the extent of proton transfer. In the case of a strong acid, such as hydrochloric acid, the acid in solution is completely ionized. In the case of a weak acid, such as acetic acid, only a small fraction of the acid in solution transfers a proton to form ions.

Equation 10.1. Strong: $HCl(aq) + H_2O(l) \rightarrow H_3O^+(aq) + Cl^-(aq)$ (~100%)

Equation 10.2. Weak: $CH_3COOH(aq) + H_2O(l) \leftrightarrows H_3O^+(aq) + CH_3COO^-(aq)$ (>5%)

For strong acids the equilibrium constant is so large that nearly all the acid is in the form of ions, and there is very little in the form of undissociated acid. For weak acids, there is an equilibrium established between the undissociated acid (reactant, on the left) and the ionized products (on the right). The extent of dissociation depends on the relative strength of the acid and is represented by the value of K_a, or **acid dissociation constant**.

Equation 10.3. $HA(aq) + H_2O(l) \leftrightarrows H_3O^+(aq) + A^-(aq)$

Equation 10.4. $$K_a = \frac{\left[H_3O^+\right]\left[A^-\right]}{\left[HA\right]}$$

The relative strength of acids can be determined by comparing their K_a values. Some typical weak acids and their respective K_a values are provided in Table 10.1. Notice that all **organic acids** (acids that have a –COOH, or carboxylic acid group) are weak acids with K_a values in the 10^{-4} to 10^{-8} range.

Table 10.1. Weak Acids and K_a Values

Name	**Formula**	**Example**	**K_a**
Formic Acid	HCOOH	Stinging ants	1.9×10^{-4}
Acetic Acid	CH_3COOH	Vinegar	1.8×10^{-5}
Benzoic Acid	C_6H_5COOH	Food preservative	6.6×10^{-5}
Lactic Acid	$CH_3CH(OH)COOH$	Sour milk	1.4×10^{-4}

The relative concentrations of hydronium (H_3O^+) and hydroxide ($OH^–$)in solution are related through the self-ionization equilibrium of water:

Equation 10.5. $2\ H_2O(l) \leftrightarrows H_3O^+(aq) + OH^-(aq)$
Equation 10.6. $K_w = 1.0 \times 10^{-14} = [H_3O^+][OH^-]$

In pure, distilled water the concentrations of hydronium and hydroxide ions are equal, with a value of 1.0×10^{-7} M. When an acid is added to water, the $[H_3O^+]$ increases and the $[OH^-]$ decreases. Conversely, when a base is added to water the $[OH^-]$ increases and the $[H_3O^+]$ decreases. Thus, one way to determine whether a solution is acidic or a basic is to measure the concentration of $[H_3O^+]$ in solution. This can be done by measuring the **pH** of the solution.

Equation 10. 7 $pH = -\log[H_3O^+]$

The pH scale ranges from 0 – 14, with pH values less than 7 being acidic, 7 being neutral, and values greater than 7 being basic.

If the $[H_3O^+]$ is $> 10^{-7}$ then the pH is < 7.00, and the solution is acidic.

If the $[H_3O^+]$ is $< 10^{-7}$ then the pH is > 7.00, and the solution is basic.

If the $[H_3O^+] = 10^{-7}$ then the pH = 7.00, and the solution is neutral.

pH of solutions can be determined using a pH meter or an indicator.

A pH meter is an electronic device that measures the $[H_3O^+]$ of a solution. It consists of an electrochemical cell with an electrode that responds to H_3O^+. It provides a relatively precise readout of pH over a broad range of pH values.

Acid-base indicators are weak acids that change color as the pH of the solution changes, based on the following equilibrium:

Equation 10.8. $HIn\ (aq) + H_2O(l) \leftrightarrows H_3O^+(aq) + In^-(aq)$

Acidic solution	Basic solution
(colorless)	(pink)

In this example the protonated indicator (HIn) is colorless in acidic solution and the unprotonated indicator (In^-) is pink in basic solution. Different indicators exhibit a characteristic color change at different pH values and can be used to estimate the pH range of a solution.

When acids and bases are added together, they participate in a **neutralization reaction** in which the acid and base properties of these substances are "neutralized." Neutralization reactions go to completion, so that all the acid and base that are added to solution react completely to form water and salt, as shown in the reaction of acetic acid with sodium hydroxide, Equation 10.6.

Equation 10.9. $CH_3COOH\ (aq) + NaOH(aq) \rightarrow H_2O(l) + CH_3COONa(aq)$

The concentration of an acid or base can be determined using a **titration** method. Titration is an analytical procedure in which one reactant is slowly added to another until the reaction is complete Typically, one of the reactants is added step-wise using a buret, this reagent is called the **titrant**, and its concentration is known. The other reactant solution is in a flask and is called the **analyte**, and its concentration is unknown. In the acid-base titration reaction between acetic acid and sodium hydroxide shown, the reaction is complete when equal moles of CH_3COOH and NaOH have been added to the reaction solution. This is called the **equivalence point** in the titration because stoichiometrically equivalent amounts of the acid and base have reacted. In other words,

Equation 10.10. # moles acid = # moles base

which can also be represented as:

Equation 10.11. $M_{acid}\ (mol/L) \cdot V_{acid}\ (L) = M_{base}\ (mol/L) \cdot V_{base}\ (L)$

If the molarity (M) and volume (V) of the titrant are known, the moles of the titrant needed to reach the equivalence point can be calculated. If the volume of the analyte sample is also known, the equation can be rearranged to determine the concentration of the analyte.

Acid-base titrations take advantage of the color change of an indicator in analyte solution or pH change of the analyte solution to determine the completion of reaction.

We can tell when the neutralization reaction is complete by adding an **indicator**, that changes color at or near the equivalence point. The point at which the indicator changes color is called the

end point of the titration. Ideally, the end point and the equivalence point should occur as close as possible in the titration.

We can also determine when the neutralization reaction is complete by monitoring the pH of analyte solution. In titration of acetic acid (analyte) with NaOH (titrant), the initial pH of analyte is less that 7 (acidic pH). As titrant is added to analyte, solution becomes more basic and pH increases. At the equivalence point moles of acid are equal to moles of base. Adding one more drop of base will change the pH so that the analyte solution becomes basic, and pH changes dramatically as illustrated in Figure 10.1. The equivalence point can be determined by plotting the second derivative of pH vs. volume.

In the first part of this experiment, you will determine the concentration of acetic acid in vinegar by titration with sodium hydroxide using phenolphthalein as the indicator to determine the *endpoint* of the reaction.

In the second part, you will use a pH meter and Logger Pro software to plot the titration curve for the neuralization reaction and to determine the *equivalence point* of the reaction. **Titration curve** is the plot of pH of analyte solution vs. the volume of titrant added. The titration of an acid by a strong base yields a typical "S" shape curve as shown in Figure 10.1. The pH of an acidic solution is initially low, and it gradually increases upon addition of the base. At the equivalence point, where the number of moles of acid are equal to the number of moles of base, there is a steep rise in pH and passed the equivalence point pH becomes constant.

Successful titrations require careful technique! Have your instructor check your apparatus and discuss appropriate technique before you begin your titrations.

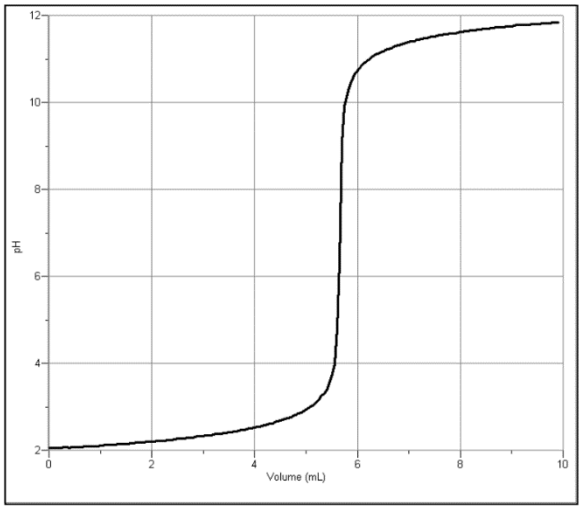


Figure 10. 1. pH vs. volume of NaOH solution.

*Name:*_____ *Acid-Base Titration*
*Section:*___ *Date:*_____

Pre-Lab Questions

1. What does it mean to say that an acid is a *strong* acid? How is a strong acid different from a weak acid?

2. What is the name of the acid present in vinegar? What is its formula? Is it a strong acid or a weak acid?

3. Write the equilibrium dissociation reaction for acetic acid and the expression for the equilibrium constant, K_a, which corresponds to this reaction.

4. Identify the weakest acid in Table 10.1. Explain your answer.

5. In a typical titration experiment a student titrates a 5.00 mL sample of formic acid with 26.59 mL of 0.1088 M NaOH. At this point the indicator turns pink. Calculate the concentration of formic acid in the original sample.

PROCEDURE

Part A: Determining the End Point Using Phenolphthalein

In this part, you will determine the concentration of acetic acid in vinegar by titration with sodium hydroxide using phenolphthalein as the indicator to determine the *endpoint* of the neutralization reaction.
Note: The endpoint is when the color of phenolphthalein in the solution changes.

1. Obtain a buret, buret clamp, a ring stand, magnetic stirrer and a magnet bar.

2. Obtain about 100 mL of standardized sodium hydroxide (NaOH) solution in a clean and dry 150 mL beaker. Record the concentration of the solution on your data sheet. Keep the solution covered until you are ready to use it in the titration.

3. Prepare the titration assembly (buret, buret clamp, ring stand, magnetic stirrer, magnet bar) as illustrated in Figure 10.2. Pour about 5 mL of titrant into the buret and rotate it so that the solution evenly coats the walls of the buret. Drain the extra solution from the buret through the stopcock. Repeat this procedure to ensure that the buret contains only sodium hydroxide at the given concentration.

4. Clean and rinse your buret and fill it with NaOH solution as instructed by the TA. Fill the buret to within 0 and 3 mL. Be sure that the tip of the buret is filled with NaOH.

5. Obtain about 20 mL of vinegar in a clean and dry 50 mL beaker. Pipet 5.00 mL of vinegar into a 250 mL Erlenmeyer flask. Add about 25 to 50 mL of distilled water—the exact amount is not important.

6. Add three drops of phenolphthalein indicator to the flask and start the stirrer at medium speed. You do not need to turn on the hot plate.

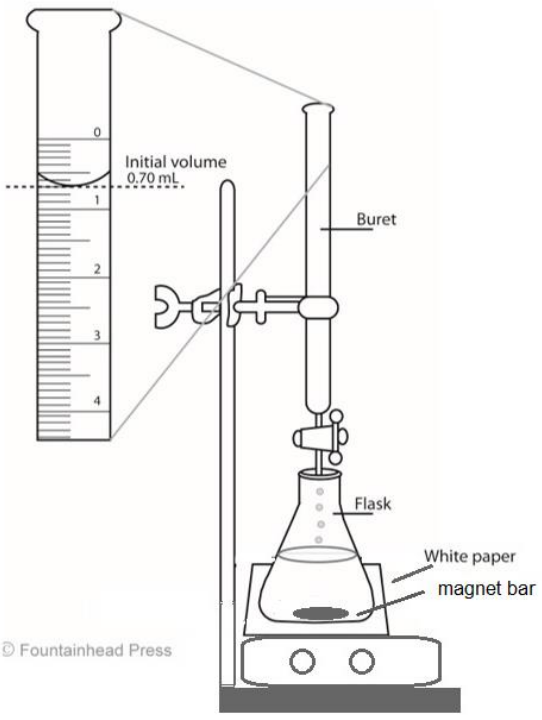


Figure 10.2. Titration setup.

7. Record the initial volume of NaOH in the buret to the nearest 0.01 mL. You may need to estimate the last digit in the volume reading. Be sure to keep your eyes at the same level as the meniscus of the solution in the buret to minimize errors in reading the buret volumes.

8. Carefully add titrant (NaOH) to the flask by opening the stopcock on the buret and allowing some NaOH to drain into the flask while stirring, as illustrated in Figure 10.3. With practice you will be able to control the stopcock with one hand while stirring the reaction mixture.

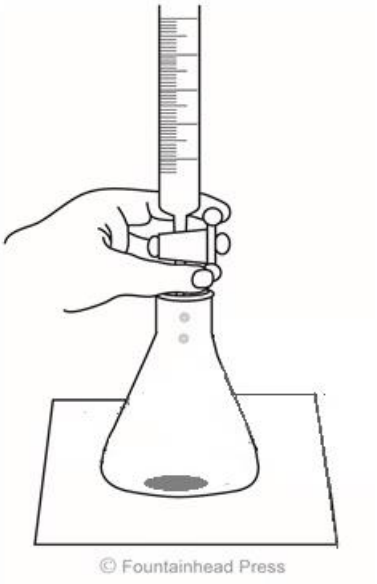


Figure 10.3. Proper titration technique.

9. Note the color of the reaction mixture as NaOH is added from the buret. As it is added to solution it will turn pink, but the pink color will disappear as the solution is mixed. As you get nearer to the equivalence point in the titration the pink color will persist longer in solution before disappearing. When this occurs, slow down the rate at which you are adding titrant.

10. As you approach the end point, add the NaOH titrant one drop at a time and wait for the solution to mix. Occasionally, wash down the sides of your flask to be sure that all of your titrant has been mixed into the reaction solution.

11. When you are very close to the end point you can "hang a drop." This is a technique for adding less than one drop by opening the stopcock slightly until a drop starts to form. Close the stopcock before the drop falls. Wash the half-drop into solution.

12. The end point in the titration is the first pink color that persists in solution after mixing. Record the volume of NaOH in the buret when you reach the end point.

13. Repeat steps 4–11 until you have completed two titrations. Refill the buret with NaOH if needed.

14. Save NaOH in the buret and vinegar in 50 mL beaker for the next part.

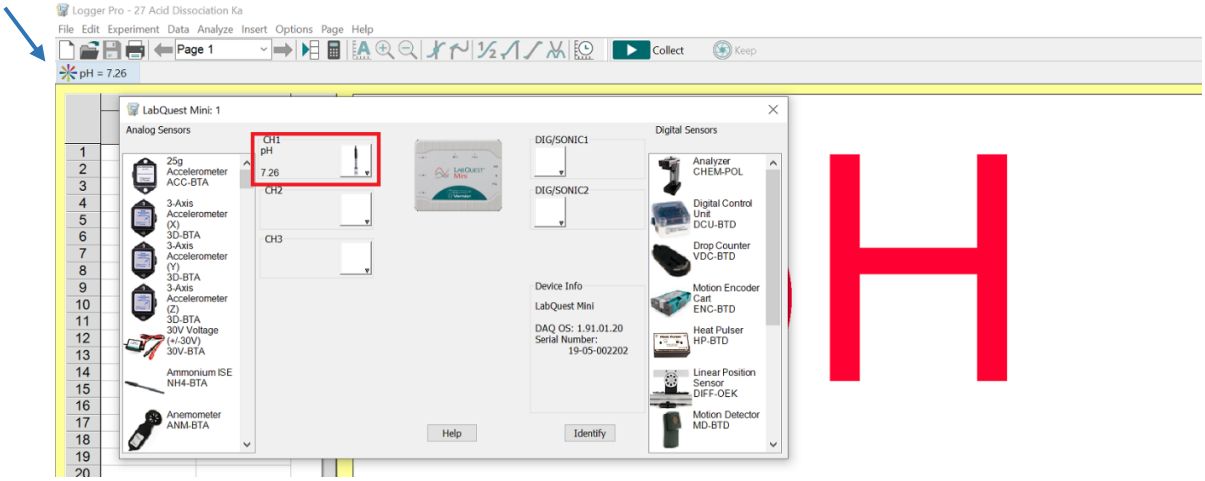
15. Discard all titration solutions in the designated waste container in the fume hood.

Part B: Determining the Equivalence Point Using pH meter

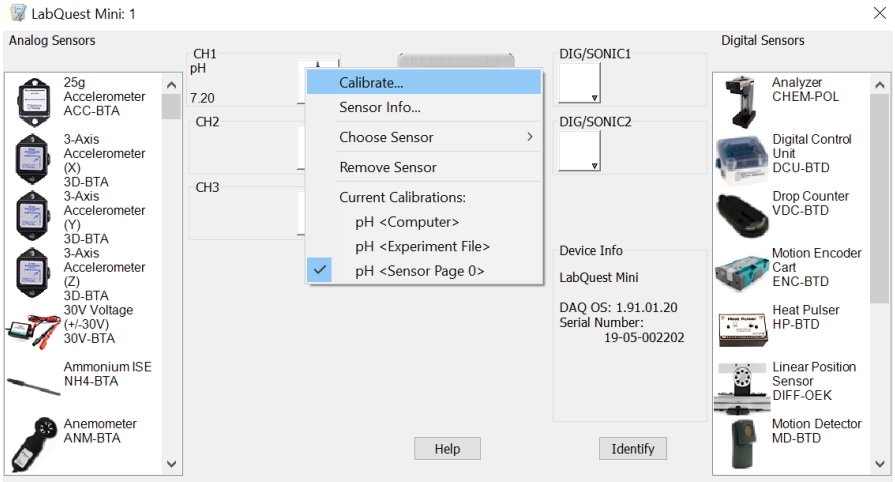
In this part, the concentration of acetic acid in vinegar will be determined using a titration curve. You will be working with a pH meter and Logger Pro software to plot the pH vs. volume of NaOH and to determine the *equivalence point* of the neutralization reaction. To begin, students need to set up and calibrate the pH meter.

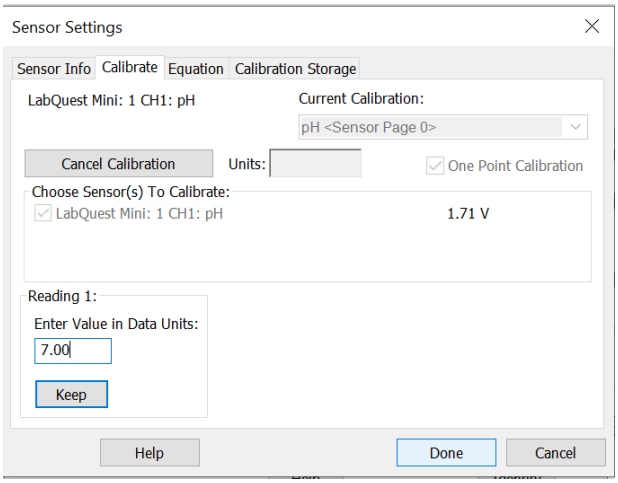
16. Connect a pH Sensor to Channel 1 of Lab Quest interface. Connect the interface to the computer using the proper cable.

17. Follow the instructions below to calibrate the pH meter.
    a. Obtain a bottle of buffer solution, pH = 7.0.
    b. Click on rainbow asterisk at the top left of the Logger Pro window. Click on the **CH1 pH** icon in the new window.



    c. Select **Calibrate** from drop-down menu.
    d. In the **Sensor Settings** box, choose **Calibrate** and **One Point Calibration.**





e. Click on **Calibrate Now**. The cursor will move down into **Reading 1** box and a voltage reading will appear above Reading 1 box.
f. Carefully immerse the pH electrode in the pH = 7.00 buffer solution. Allow the pH reading (voltage) to stabilize.
g. When the voltage is stabilized, enter **7.00** in the **Reading 1** box.
h. Click **Keep** and **Done**.
i. Close the dialog box.
j. If the displayed pH reading is 7.00 you have successfully calibrated the sensor. You are ready to begin the experiment. If the reading is not 7.00, repeat calibration.

15. Remove the pH probe from the buffer solution, rinse with deionized water and gently pat dry. **You must rinse and dry the pH probe after each pH measurement.**

16. Close the buffer solution bottle cap and save for the next group.

17. After the calibration is completed, prepare for the titration and data collection. Pipet 5.00 mL of vinegar into a 150 mL beaker. Add about 25 to 50 mL of distilled water—the exact amount is not important.

18. Place the beaker on a hot plate and add a stirring bar into the beaker. Do NOT turn on the heat!

19. Use a clamp to suspend the pH senso on the hot plate, as shown in Figure 10.4.

20. Position the pH sensor so that its tip is immersed in the vinegar solution but is not struck by the stirring bar. Gently turn on the stirrer knob on the hot plate, you may need to move the beaker a little bit to adjust the position of the stirring bar.

21. Open the Logger *Pro* folder on your computer. Open the file **"07a Acid-Base"** from the **Advanced Chemistry with Vernier** folder.

22. To begin the experiment, press **Collect** in the ribbon menu.
    a. Once the displayed pH reading has stabilized, click **Keep**. In the edit box, type **0** (for 0 mL added). Click **OK** to continue.

b. Carefully open the stopcock on buret and add 2 mL of NaOH. Wait for pH reading to stabilize and click **Keep**. Enter the volume of added NaOH as accurately as possible in the popup menu and click **OK** to continue.

   Note: Adding NaOH to HCl occurs in three stages, the first phase is up to approximately pH 6, the second phase is up to approximately pH 10, and the third phase is at pH levels above 10.

c. Carefully add a small amount of NaOH that increases the pH by approximately 0.10 units. Close the stopcock, click **Keep**, enter the ***total volume*** of NaOH in the edit box.

   Note: Initially, addition of 2-3 mL of NaOH leads to an increase in pH by approximately 0.10 units. As the equivalence nears, a single drop of NaOH can cause a pH shift of around 0.10 units. After reaching the equivalence point, the pH becomes stable and undergoes minimal changes. As NaOH is added, keep your hand on the stopcock and watch the pH change from live readout.

d. Continue adding NaOH slowly to make the pH change by about 0.10 unit. Once a pH value of approximately 6.0 is achieved, switch to adding NaOH in two-drop increments, where 1 drop is equivalent to approximately 0.050 mL and 2 drops is about 0.10 mL.

e. Once a pH value of approximately 10 is achieved, switch to adding 2-3 mL increments of NaOH. At this point, the pH will change by about 0.10 units. Remember to enter the ***total volume*** of added NaOH after each addition.

f. Continue adding NaOH solution until the pH value remains constant for three to five consecutive measurements.

23. You may need to rescale the axes to see all the data points. Ask your TA for help if needed.

24. When you have finished collecting data, click **Stop**.

25. Rinse the pH probe with distilled water, gently dry and place in buffer solution.

26. Follow the steps below to find the *equivalence point*, which is the largest increase in pH upon the addition of a very small amount of NaOH solution. An efficient method of determining the precise equivalence point of the titration is to plot the ***second derivative*** of the pH versus volume data. The equivalence point can be more easily observed in a second derivative graph as it intersects the x-axis.

    a. To view the second derivative plot, click the **Next Page** on top of the data table to go to **Page 3**.

    b. The intersection of the second derivative plot with the x-axis indicates the equivalence point. Determine and record the volume of NaOH at the equivalence point by positioning the cursor on the second derivative plot, as shown in Figure 10.5.

27. Save a copy of each graph by taking a screen shot. You will need to submit these graphs along with your report.

    Note: Click **Cancel** if a **Print Setup** menu appears. Choosing a printer from this menu may cause the software to become nonresponsive.

28. Exit the software. Do not save.

29. Discard all titration solutions in the designated waste container in the fume hood.

30. Rinse the buret three times with tap water and two times with deionized water. Open the stopcock each time so that the tip is rinsed too.

31. Store the buret upside down on the stand with the stopcock in open position. This prevents the clogging of the stopcock and the tip.

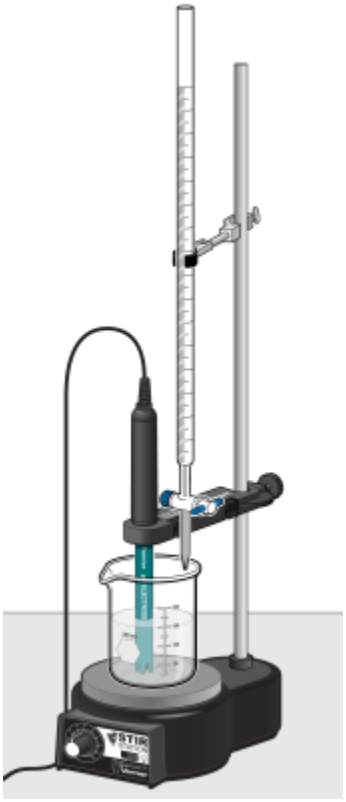


Figure 10. 4. Titration setup with pH meter.

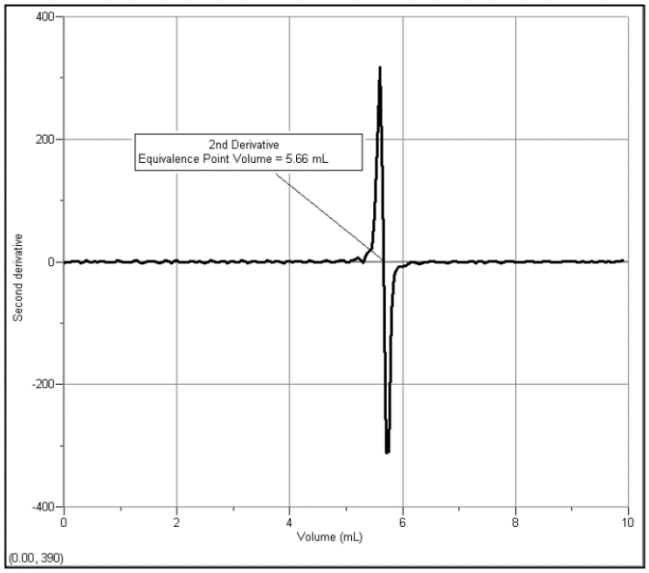


Figure 10. 5. Second derivative of pH-volume data.

CALCULATIONS

Show all work on your data sheet:

Part A: Determining the End Point Using Phenolphthalein

1. For each trial, subtract the initial NaOH volume from the final volume to obtain the total NaOH volume used in each titration. This volume should be reported to the nearest 0.01 mL.

2. Using the molarity and volume of the NaOH solution used for each trial, calculate the moles of sodium hydroxide added for each trial. Record this value on your data sheet for each trial. Be sure to convert your volume from mL to liters before calculating. For example, if you used 35.27 mL of 0.1055 M NaOH, the moles of NaOH would be calculated as:

   $$\text{moles NaOH} = M_{NaOH} \cdot V_{NaOH} = (0.1055 \text{ mol/L})(0.03527 \text{ L}) = 3.721 \times 10^{-3} \text{ mol NaOH}$$

   Be sure to record your results to the appropriate number of significant figures.

3. Since the stoichiometry of the neutralization reaction is 1:1, the moles of acetic acid in your 5.00 mL vinegar sample equals the moles of NaOH needed to reach the end point.

   $$\#\text{ moles acetic acid} = \#\text{ moles NaOH}$$

Record the moles of acetic acid for each trial on your data sheet.

4. The molarity of acetic acid in the vinegar sample can be calculated by dividing the moles of acetic acid by the volume of the vinegar sample (5.00 mL, or 0.00500 L).

   $$M_{acetic\ acid} = \frac{\#\ moles\ acetic\ acid}{0.00500\ L}$$

Record the molarity for each trial on your data sheet.

Part B: Determining the Equivalence Point Using pH meter

5. Use the 2nd derivative plot of pH-volume data to determine the volume of NaOH at the equivalence point.
6. Repeat steps 3 and 4 to calculate the concentration of acetic acid in vinegar.

*Name:*_____ *Acid-Base Titration*
*Section:*__ *Date:*_____

Titration of Vinegar
Data Sheet

Part A: Determining the End Point Using Phenolphthalein

Titrant: _____ M NaOH

Sample: _____ mL vinegar

	Trial 1	Trial 2
Final volume (mL):	_____	_____
Initial volume (mL):	_____	_____
Volume NaOH Delivered (mL):	_____	_____
Moles of NaOH:	_____	_____
Moles acetic acid:	_____	_____
Molarity of acetic acid:	_____	_____

Average Molarity: _____

Show sample calculations.

*Name:*_____ *Acid-Base Titration*
*Section:*___ *Date:*_____

Titration of Vinegar
Data Sheet

Part B: Determining the Equivalence Point Using pH meter

Titrant: _____ M NaOH

Sample: _____ mL vinegar

Volume NaOH at equivalence point (mL): _____
(from the 2nd derivative plot)

Moles of NaOH: _____

Moles acetic acid: _____

Molarity of acetic acid: _____

Attach your graphs.

Show your calculations.

*Name:*_____ *Acid-Base Titration*
*Section:*__ *Date:*_____

Post Lab Questions

1. Why do you rinse the buret with the sodium hydroxide solution and not with distilled water? How would your titration results be affected if you rinsed with distilled water before filling your buret? (Hint: how would the actual molarity of NaOH in the buret be affected?)

2. In step 4 of the procedure you were told to add between 25–50 mL of distilled water, but that the actual volume was not important. Explain why the volume of water you add in this step is not important.

3. Explain the difference between the equivalence point and the end point.

4. Which of the two methods in this experiment will yield more accurate results for concertation of acetic acid in vinegar? Explain.