DETERMINATION OF ACETIC ACID IN VINEGAR

STANDARDIZATION OF NaOH (OPT.)

Introduction:

Vinegar is a dilute solution of acetic acid. Since vinegar is an acid it can be titrated with a base. Titration is the process of adding a known amount of a solution of known concentration to a known amount of solution of unknown concentration. The more accurately the concentration of the solution of known concentration is known, the more accurately the concentration of the unknown solution can be determined. Some chemicals can be purchased in a pure form and remain pure over a long period or time. Other chemicals are easily contaminated by the absorption of carbon dioxide or water from the air. Sodium hydroxide absorbs moisture from the air and often appears wet. Thus if a solution of sodium hydroxide is prepared by weighing the sodium hydroxide, the concentration of the solution may not be precisely the intended concentration. Potassium hydrogen phthalate on the other hand, has less of a tendency to absorb water from the air and when dried will remain dry for a reasonable period of time. Potassium hydrogen phthalate may be purchased in pure form at reasonable cost. Potassium hydrogen phthalate is a primary standard. This means that carefully prepared solutions of known concentration of potassium hydrogen phthalate may be used to determine, by titration, the concentration of another solution such as sodium hydroxide. The equation for the reaction of potassium hydrogen phthalate with sodium hydroxide is:

$$KCO_2C_6H_4CO_2H + NaOH \rightarrow KCO_2C_6H_2CO_2Na + H_2O$$

The equivalence point of a titration occurs when chemically equivalent amounts of acid and base are present. At this point the pH changes rapidly with a small addition of acid or base. If a pH meter is used in the titration and the pH plotted vs the volume of base added, the equivalence point is the middle of the vertical part of the curve. Once the equivalence point of the titration is known, the concentration of the sodium hydroxide can be determined. The sodium hydroxide is then used to titrate vinegar, and the concentration of the vinegar is determined. The percent acetic acid can be determined from the concentration of the vinegar.

Purpose:

The purpose of this laboratory activity is to determine the percent acetic acid in a commercial vinegar sample by the method of standardization of a solution.

Equipment/Materials:

pH meter	buffer pH 7 & 10
NaOH solution	vinegar
droppers	stirrer and stir bar
buret	buret clamp
beaker (100, 250 mL)	pipet 10mL
funnel	analytical balance
graph paper or graphing program	potassium hydrogen phthalate (KHP)
scoopula	

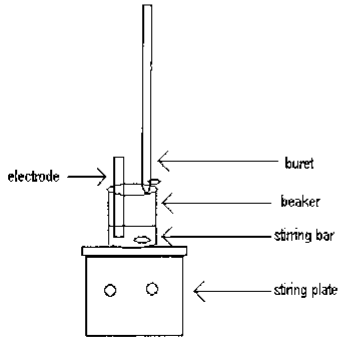
Safety:

- Always wear apron and goggles in lab
- The NaOH is especially dangerous. Use caution when handling it.

Procedure Part I: Standardization of NaOH (opt.)

1. Plug in the pH meter and allow it to warm up for about 10 munites.
2. The temperature knob should be set between 20 and 25°C.
3. Remove the cap from the electrode, and rinse the electrode with DI water. Blot the end with a Kimwipe.
4. Place the electrode in the pH 7 buffer, turn the knob to pH, and adjust the pH to 7.00 with the standardization knob.
5. Place the instrument on standby. Rinse and blot the electrode.
6. Place the electrode in pH 10.00 buffer, turn the knob to pH, and adjust the pH to 10.00 with the slope knob.
7. Place the instrument on standby. Rinse and blot the electrode.
8. Rinse the buret with ~ 5mL of NaOH solution.
9. Rinse and fill the buret past the 0-mL mark with NaOH solution.
10. Drain some of the NaOH through the tip into a waste beaker to fill the tip and bring the level of NaOH to exactly 0.00 mL. If the 0.00 mL mark is passes, use a dropper to bring the meniscus to 0.00 mL.
11. Weigh about 0.3 g of potassium hydrogen phthalate. Record the exact mass of the potassium hydrogen phthalate.

12. Place the potassium hydrogen phthalate in a beaker.
13. Add ~ 25 mL of distilled water and dissolve the potassium hydrogen phthalate.
14. Position the magnetic stirrer so that the buret and pH electrode may both be placed in the 250-mL beaker when it is on the stirrer.
15. Place the beaker on the stirrer and the stirring bar. . Position the electrode so that the bulb is in the solution but above the stir bar. Add more distilled water if necessary. See diagram.



16. Record the initial pH for 0.00 mL of NaOH added.
17. Add NaOH until the pH has increased by ~0.2 pH units.
18. Record the **pH** and the buret reading to **two** decimal places.
19. Repeat steps 17-18 until a pH of 12 is obtained or until the buret reading = 50.00 mL,. Do not allow the NaOH to go below 50.00 on the buret.
20. Repeat the procedure for other trials as instructed.
21. Graph the pH (y) vs volume NaOH (x).

Procedure Part II: Determine the % acetic acid in Vinegar.

1. Plug in the pH meter and allow it to warm up for about 10 munites.
2. The temperature knob should be set between 20 and 25°C.
3. Remove the cap from the electrode, and rinse the electrode with DI water. Blot the end with a Kimwipe.
4. Place the electrode in the pH 7 buffer, turn the knob to pH, and adjust the pH to 7.00 with the standardization knob.
5. Place the instrument on standby. Rinse and blot the electrode.
6. Place the electrode in pH 4.00 buffer, turn the knob to pH, and adjust the pH to 4.00 with the slope knob.
7. Place the instrument on standby. Rinse and blot the electrode.
8. Place a clean 250-mL beaker on the balance and tare.
9. Using a pipet place exactly 10.00 mL of vinegar in the 250-mL beaker. Record the mass of the vinegar.

10. Add ~ 75 mL of distilled water.
11. Refill the buret to 0.00 mL. See steps 9-10 in Part I.
12. Position the magnetic stirrer so that the buret and pH electrode may both be placed in the 250-mL beaker when it is on the stirrer.
13. Place the beaker on the stirrer and add the stirring bar. Position the electrode so that the bulb is in the solution but above the stir bar. See diagram in Part I.
14. Record the initial pH for 0.00 mL of NaOH added.
15. Add NaOH until the pH has increased by ~ 0.2 pH units.
16. Record the pH and the buret reading to **two** decimal places.
17. Repeat steps 14-15 until a pH of 12 is obtained or until the buret reading = 50.00 mL, Do not allow the NaOH to go below 50.00 on the buret.
18. Repeat the procedure for other trials as instructed.
19. Graph the pH (y) vs volume NaOH (x).

Name _____

Name _____

Period _____

Date _____

ACETIC ACID IN VINEGAR

Data Table: Part I (opt.)

Mass of potassium acid phthalate used _____g

Trial Number _____

Vol. NaOH	PH

Vol. NaOH	pH

Volume of NaOH at end point _____ mL

Data Table: Part II

Mass of vinegar _____ g

Trial Number _____

Vol. NaOH	PH

Vol. NaOH	pH

Volume of NaOH at end point _____ mL

Moles of NaOH at end point _____ mol

Moles of vinegar neutralized _____ mol

Mass of acetic acid in vinegar _____ g

Percent of acetic acid in vinegar _____ %

Calculations: Part I (opt.)

1. Mark the end point of the titration on the graph. From the graph determine the volume of NaOH needed to reach the equivalence point.

2. Calculate the number of moles of potassium hydrogen phthlate used.

3. Calculate the number of moles of NaOH needed to reach the equivalence point.

4. Calculate the concentration of the NaOH solution

Calculations: Part II

1. Write the balanced equation for the neutralization reaction and calculate the number of moles of vinegar neutralized.

2. Calculate the number of grams of acetic acid in the vinegar.

3. Calculate the percent of acetic acid in vinegar.
   Percent acetic acid = (mass of acetic acid/mass of vinegar) x 100.

Questions:

1. Why was the buret rinsed with NaOH?

2. What would be the affect on the percent acetic acid if 30 mL of vinegar had been used in place of 10 mL? Explain?

3. Why was the potassium hydrogen phthalate used?

ACETIC ACID IN VINEGAR
TEACHER NOTES

Lab Time:

Part I: 45 minutes
Part II: 45 minutes

Preparations:

Time: 35 minutes

Dry the KHP for 1 hour at 110 °C and store in a desiccator.

Turn on and warm up the pH meters 15 minutes before class.

Prepare 0.25 M NaOH by dissolving 10.0 g of NaOH in enough freshly boiled distilled water to give a volume of 1 L.

Answers to Questions:

1. Why was the buret rinsed with NaOH?
   To remove any water or other substances that might have been in the buret.

2. What would be the affect on the percent acetic acid if 30 mL of vinegar had been used in place of 25 mL? Explain?
   There would be no change in % acetic acid. It would take more NaOH to reach the end point.

3. Why was the potassium hydrogen phthalate used?
   To determine the concentration of the NaOH accurately..

Considerations:

Time can be reduced by having the pH meters standardized and/or filling the burets with NaOH and having burets, pH meters and stirrers in position. The KHP standardization and vinegar titration should be completed on the same day since NaOH tends to change concentration slightly on standing. Time can be reduced by having each group of students do fewer trials and then sharing class data to determine a final average concentration of vinegar.

The H. J. Heinz Company quality control on vinegar is 4.98 to 5.03%. They ship nothing under 5%.

Determination of the Ionization Constant of a Weak Acid

PURPOSE: To determine the pK_a and K_a of the weak monoprotic acid, acetic acid.

BACKGROUND: In this experiment, a weak monoprotic acid will be titrated with a solution of the strong base NaOH. The pH will be monitored by a computer interfaced pH electrode and the volume of the NaOH solution will be entered from the keyboard with each addition of the base to the acid solution. A plot of pH on the vertical axis versus volume of NaOH on the horizontal axis gives a typical weak acid-strong base titration curve. At the point on the curve where the amount of base added is equal to one-half of the amount of base needed to reach the equivalence point (end point), the concentrations of the remaining acid, HA, and its ion, A^-, are equal. At this point the pH is equal to the pK_a. This can be explained by taking a closer look at the equilibrium involved in the ionization of a weak acid.

For a weak acid, HA, the ionization equilibrium and the equilibrium expressions are

$$HA + H_2O \leftrightarrow H_3O^+ + A^- \qquad \text{Eqn. 1}$$

$$K_a = \frac{[H_3O^+][A^-]}{[HA]} \qquad \text{Eqn. 2}$$

This expression can be arranged to get

$$[H_3O^+] = K_a\left(\frac{[HA]}{[A^-]}\right) \qquad \text{Eqn. 3}$$

Taking the -log of this expression gives

$$-\log [H_3O^+] = -\log K_a - \log\left(\frac{[HA]}{[A^-]}\right) \qquad \text{Eqn. 4}$$

Since $pH = -\log [H_3O^+]$ and $pK_a = -\log K_a$, substitution gives

$$pH = pK_a - \log\left(\frac{[HA]}{[A^-]}\right) \qquad \text{Eqn. 5}$$

(Now this is the important part.) At the midpoint of a titration (i.e. when exactly half of the base needed to reach the end point of a titration has

been added) half of the HA has been converted to A^-. "So?" you might ask. This means that [HA] = [A^-]. Since ([HA]/[A^-]) = 1 and the log of 1 = 0, then equation 5 merely becomes **pH = pK_a.** **The -antilog pK_a = 10^{-pKa} = K_a.**

PROCEDURES

Part I: Computer Setup

1. **First,** turn on the *Science Workshop 500* interface. The power switch is on the back. The green light on the front indicates it is on.
2. Turn on the computer.
3. Press Ctrl-Alt-Delete when the 'Welcome to Windows' window appears.
4. Click 'OK' in the 'Log On to Windows' window appears.
5. Double click on 'Data Studio.'
6. Click on 'Open Activity.'
7. Double click the file'C32 Titration.'
8. Close 'Titration Workbook' window.
9. Drag 'pH and Volume Table' window to the right side of the screen.
10. Drag 'pH Digits' window to the bottom of the screen.

Part II: Sensor Calibration and Equipment Setup

- To calibrate the pH sensor you will need a 250 mL beaker half-filled with distilled water, pH 4.0 buffer solution, and pH 9.2 buffer solution.

1. Click the 'Setup' button.
2. Double click on 'pH' beneath the picture of the interface box. The 'Sensor Properties' window will appear.
3. Click on the 'Calibration' tab.
4. Calibrate with the 9.18 buffer solution
   - Rinse the pH electrode in the beaker of distilled water.
   - Put the pH electrode in the buffer solution.
   - Check the voltage under 'Current Reading.'
   - When the 'Current Reading' voltage stabilizes, click the 'Take Reading' button under 'High Point'.
   - Delete whatever value is in the 'Value' box and enter the pH value of 9.2.
5. Rinse the pH electrode in the beaker of distilled water.
6. Calibrate the low pH buffer solution.
   - Put the pH electrode in the pH 4.0 buffer solution.
   - Check the voltage under 'Current Reading.'

- When the 'Current Reading' voltage stabilizes, click the 'Take Reading' button under 'Low Point'.
- Delete whatever value is in the 'Value' box and enter the pH value of 4.0.

7. Click 'OK'. This closes the 'Sensor Properties' window.
8. Place the pH electrode in the beaker of distilled water.
9. Close the 'Experiment Setup' window.

Part III: Equipment Setup

1. After rinsing a 50-mL buret with three 5 mL portions of 0.10 M NaOH solution, fill the buret with 0.10 M NaOH.
2. Place a beaker to collect waste solution under the buret and open the stopcock to force any air out of the buret tip. Make sure that the buret tip is filled with NaOH. Set the initial buret reading to the 0.00mL gradation line.
3. Carefully place a stir bar into a clean, dry 150-mL beaker.
4. Add 25.00 mL of 0.10 M acetic acid to the beaker.
5. Place the beaker with the acid and stir bar on the magnetic stirrer and underneath the buret so that the tip of the buret is above the acid solution.
6. Place the pH electrode in the acid solution being careful not to have the stir bar hit the electrode. Turn on the stirrer.
7. Optional: Add 2 drops of phenolphthalein to the beaker.

Part IV: Data Recording

- Note: Data recording goes faster if one person controls the buret value and reads the volume of titrant added to the acid while a second person operates the computer and enters the volumes.

1. Click 'Sampling Options' in the 'Experimental Setup' window.
2. Check (click) the 'Prompt for a value' under the 'Manual Sampling' tab.
3. Close the 'Sampling Options' dialog box and the 'Experimental Setup' window.
4. Click 'Start.' Note that the 'Start' button has changed to a 'Keep' button and that the timer is now running. Ignore the timer.
5. Click 'Keep' and type "0" for the volume of NaOH added to the HAc. Then click 'OK.'
6. Add 1.0 mL NaOH to the HAc. Allow the pH to stabilize, then click 'Keep.' Type "1.0" and click 'OK.'

7. Add another mL of NaOH, let the pH stabilize, then click 'Keep.' Record the new volume added when prompted to do so. Repeat this procedure until the upward slope is approached. **As you continue toward the endpoint, reduce the volume of NaOH added to a few drops at a time.** Continue to add the NaOH slowly until the slope of the curve begins to plateau, at which time you may again add the titrant in 1-mL portions.
8. Stop recording data after five consecutive 1-mL additions of NaOH yields no change in pH (or until you have added 50.00 mL of NaOH).
9. Use the Smart Tool to determine the volume of NaOH added at the endpoint, the volume of NaOH added to reach the midpoint, and the pH at the midpoint. Record these values. Print the graph.*
10. Perform a second run, switching roles; i.e., the person operating the computer should now work with the buret. Analyze the graph using the Smart Tool as you did in step 10.
11. When data recording stops, turn off the magnetic stirrer. Remove the pH sensor from the titrated acid solution, rinse it with distilled water, and place it back in the 'pH 4.01' buffer solution.
12. Decant the solution in the beaker down the drain **being careful to save the stir bar.** Rinse the beaker with tap water and remove the stir bar before dumping the rinse water down the drain.

* You might also determine the endpoint of the titration by graphing the second derivative. See the supplemental instructions.

Part V: Working with the Graph and Calculations

1. Print out a copy of your graph (one per person).
2. Find the point on the graph line that represents the endpoint of the titration. This is the inflection point of the curve and is also the endpoint of the titration. Neatly label this position the 'Endpoint' on your print out.
3. **Use a straight edge** to extend a vertical line from the endpoint down to the x-axis. This represents the volume of NaOH added at the endpoint of the titration.
4. Divide the volume from step 3 by 2. This represents the volume of NaOH added at the midpoint of the titration. Find this volume on the x-axis.

5. Again, using a straight edge, draw a vertical line up from the volume of NaOH added at the midpoint of the titration to the graph line. Neatly label this point on the line as the 'Midpoint.'
6. Use the straight edge to extend a horizontal line from the midpoint to the y-axis. This is the pH of the solution in the beaker at the midpoint of the titration.
7. use the 'Smart Tool' to help you approximate the value of the pH at the midpoint **and record this value at the bottom of the graph.** Hand written is OK.
8. Show your calculations for the K_a at the bottom of the graph. Hand written is OK.

DATA TABLE (for formal report):

Volume of NaOH added at the endpoint (mL)	
Volume of NaOH added at the midpoint (mL)	
pH of the solution at the midpoint	
pK_a of the solution at the midpoint	
K_a	

CALCULATIONS:

If, and only if, the pH is taken at the midpoint of a titration does the pH = pK_a.

$pH = pK_a$ and the -antilog $pK_a = 10^{-pKa} = K_a$

Steps to produce graphs of the first and second derivatives of pH vs. Volume graph.

For the first derivative:

1. Click 'Calculate'
2. Delete 'y = x' under 'Definition'
3. Type "slope1 = "
4. Click on 'Special' tab.
5. Click 'derivative(2,x)'
6. Click the down arrow under 'Variables:'
7. Click 'Data Measurement'
8. Click 'Run #1' under 'pH, ChA vs Volume (pH)
9. Click 'OK'
10. Click 'Accept'
11. In the Data Summary window click and drag 'Run #1' [under 'slope1 = derivative(2,x)] to 'Graph' on the Displays window.
12. The graph of the first derivative will appear.

To produce a graph of the second derivative:

1. Click 'Calculate'
2. Click 'New'
3. Type "slope2 = derivative(2,slope1)"
4. Click the down arrow under 'Variables:'
5. Click 'Data Measurement'
6. Click 'Run #1' under 'slope1 = derivative(2,x)
7. Click 'OK'
8. Click the down arrow under 'Variables:' [It now says 'Define slope1.]
9. Click 'Data Measurement'
10. Click 'slope1 = derivative(2,x)
11. Click 'OK'
12. Click 'Accept'
13. In the Data Summary window click and drag 'Run #1' under 'slope2 = derivative(2,slope1)' to 'Graph' in the Displays window.
14. The graph of the second derivative should now appear.
15. The volume of the titrant added at the endpoint of the titration is represented by the value of the volume at the x-intercept.