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 of 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 a lesser 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:

 $\text{KCO}_2\text{C}_6\text{H}_4\text{CO}_2\text{H} + \text{NaOH} \rightarrow \text{KCO}_2\text{C}_6\text{H}_4\text{CO}_2\text{Na} + \text{H}_2\text{O}$

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.

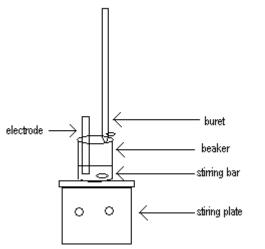
Equipment/Materials:

pH meter	buffer pH 7 & 10
0.25M 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 scoopula	potassium hydrogen phthalate (KHP)
Safety:	

- Always wear apron and goggles in the 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 minutes.
- 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 \sim 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 passed, 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.

- 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 minutes.
- 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 \sim 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).

Data Table: Part I (opt.)

Mass of potassium acid phthalate used _____g

Trial Number _____

Vol. NaOH	PH

Vol. NaOH	nH
	рп

Volume of NaOH at end point



Data Table: Part II

Mass of vinegar _____ g

Trial Number _____

Vol. NaOH	РН

Vol. NaOH	рН

Volume of NaOH at end point Moles of NaOH at end point Moles of vinegar neutralized Mass of acetic acid in vinegar Percent of acetic acid in vinegar

	_mL
	mol
	_mol
	_g
	_%

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 phthalate 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. From a graph of your data, determine the volume of NaOH at the equivalence point.
- 2. Write the balanced equation for the neutralization reaction, and calculate the number of moles of vinegar neutralized.
- 3. Calculate the number of grams of acetic acid in the vinegar.
- 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 effect 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?