

## Experiment 15

FV 09/13/10

### ANALYSIS OF VINEGAR

**MATERIALS:** Four 250 mL Erlenmeyer flasks, two 50 mL burets, liquid funnel, one 150 mL beaker, one 50 mL beaker, 5.00 mL pipet, weighing bottle, weighing dish, approximately 0.1 M NaOH, C<sub>6</sub>H<sub>4</sub>(COOH)COOK (potassium hydrogen phthalate), phenolphthalein, grease pencil.

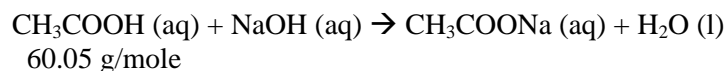
**PURPOSE:** The purpose of this experiment is two-fold: (1) to standardize a solution of sodium hydroxide; (2) to use the standardized sodium hydroxide solution to titrate the acetic acid in vinegar to determine its percent by mass in vinegar.

**LEARNING OBJECTIVE:** By the end of this experiment, the midshipman should be able to demonstrate the following proficiencies:

1. Use pipets and burets correctly.
2. Explain the term hygroscopic.
3. Explain the function and necessary qualities of a primary standard.
4. Perform a titration accurately and precisely.
5. Calculate the molarity and the mass of components in a solution from stoichiometry information.
6. Calculate the percent by mass of a component in a solution.

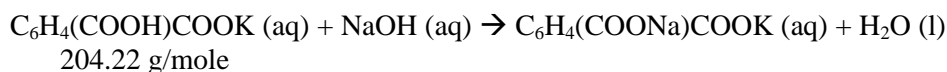
#### DISCUSSION:

Vinegar is a solution of acetic acid in water. Acetic acid, CH<sub>3</sub>COOH, is a weak monoprotic acid with a molar mass of 60.05 g/mole. The percent by mass of acetic acid in vinegar can be determined by titrating a known amount of vinegar with a *standardized* solution of sodium hydroxide, i.e., a sodium hydroxide solution of accurately known concentration. Acetic acid and sodium hydroxide react as shown below:



Sodium hydroxide is a *hygroscopic* solid which means it absorbs water from the air. A weighed quantity of sodium hydroxide therefore contains an unknown mass of water. Therefore, a solution of known molarity cannot be made by dissolving a known mass of solid sodium hydroxide in water. The concentration of a sodium hydroxide solution must be determined experimentally. This is done by titrating the sodium hydroxide solution against a *primary standard*. A primary standard is a substance from which a solution of known concentration can be prepared.

The primary standard used in this experiment is potassium hydrogen phthalate, C<sub>6</sub>H<sub>4</sub>(COOH)COOK, which is referred to by the shorthand notation of "KHP". KHP has several advantages: it does not absorb moisture readily (i.e., is not hygroscopic); it is easily dried; it can be accurately weighed; it can be obtained in very pure form; it has a high molar mass of 204.22 g/mole; and it is very soluble in water. KHP is an acid, which reacts in aqueous solution to neutralize the base, sodium hydroxide, as shown below:



## PROCEDURE:

### Part A. Standardization of the Sodium Hydroxide Solution

1. Use the top-loading balance to pre-weigh about 0.51 grams of KHP into a weighing dish.
2. Use the analytical balance to obtain the mass of the weighing dish and the KHP sample. *Quantitatively transfer* the KHP to a clean 250 mL Erlenmeyer flask. (A qualitative transfer is a complete transfer of the sample between containers with no spills.) Obtain the mass of the empty weighing dish.
3. Add approximately 100 mL of distilled water to the flask to dissolve the KHP and then add two to three drops of phenolphthalein indicator. Gently swirl the solution until all of the KHP is dissolved.
4. Add ~ 50 mL of NaOH solution (approximately 0.1 M) to a 150 mL beaker. Pour the NaOH solution from the beaker, through a liquid funnel, into the buret. Make sure there are no air bubbles in the buret tip. Record your initial buret reading with the proper significant figures.
5. Titrate the KHP solution with the NaOH solution, to a very pale pink endpoint. Record the final buret reading.
6. Repeat this process. If two titrations are not within +/- 0.50 mL of each other, repeat this process again.

### Part B. Measurement of Acetic Acid in Vinegar

1. Use the analytical balance to obtain the mass of a clean, empty weighing bottle with cap. It is ok if the bottle is a little wet inside as long as it is only distilled water. It must be dry on the outside.
2. Add ~ 15 mL of vinegar to a 50 mL beaker. Pre-rinse a 5.00 mL pipet with a small quantity of vinegar. Pipet a 5.00 mL sample of vinegar into the weighing bottle and cap the bottle. Reweigh the bottle containing the 5.00 mL sample.
3. *Quantitatively transfer* the vinegar sample, with rinses, to a clean 250 mL Erlenmeyer flask.
4. To the vinegar sample in the 250 mL Erlenmeyer flask, add approximately 100 mL of distilled water and then two or three drops of phenolphthalein indicator.
5. Refill your buret with the NaOH solution.
6. Titrate the vinegar solution with the NaOH solution, to a very pale pink endpoint.
7. Repeat this process. If two titrations are not within +/- 0.50 mL of each other, repeat the titration.

### Clean up:

1. Rinse all waste solutions down the drain.
2. Wash all glassware. Invert the buret and mount it in the buret clamp on the ring stand, with the stopcock open to allow it to drain. Leave the other washed glassware upright. Clean up your area.

Name \_\_\_\_\_

Section \_\_\_\_\_

Partner \_\_\_\_\_

Date \_\_\_\_\_

**DATA SECTION**  
**Experiment 15**

INCLUDE THE APPROPRIATE SIGNIFICANT FIGURES.

**Part A. Standardization of the Sodium Hydroxide Solution**

	Sample 1	Sample 2	Sample 3
Mass of weighing dish + KHP (g)			
Mass of weighing dish (g)			
Mass of KHP (g)			
Initial buret reading of NaOH (mL)			
Final buret reading of NaOH (mL)			
Volume of NaOH used (mL)			

Need 2 good titrations within +/- 0.50 mL

**Part B. Measurement of Acetic Acid in Vinegar**

	Sample 1	Sample 2	Sample 3
Mass of weighing bottle with cap (g)			
Mass of weighing bottle with cap + 5.00 mL Vinegar (g)			
Mass of 5.00 mL Vinegar (g)			
Initial buret reading of NaOH (mL)			
Final buret reading of NaOH (mL)			
Volume of NaOH used (mL)			

Need 2 good titrations within +/- 0.50 mL

**DATA TREATMENT**  
**Experiment 15**

INCLUDE THE APPROPRIATE SIGNIFICANT FIGURES.

**Part A. Standardization of the Sodium Hydroxide Solution**

(A.1) Calculate the moles of KHP for:

Sample 1:

Sample 2:

Sample 3:

(A.2) Calculate the moles of NaOH for:

Sample 1:

Sample 2:

Sample 3:

(A.3) Calculate the concentration (M) of the NaOH solution for each sample, and then obtain the average value:

Sample 1:

Sample 2:

Sample 3:

AVERAGE:

**DATA TREATMENT continued**  
**Experiment 15**

**Part B. Measurement of Acetic Acid in Vinegar**

(B.1) Calculate the moles of NaOH for:

Sample 1:

Sample 2:

Sample 3:

(B.2) Calculate the moles of acetic acid in 5.00 mL vinegar:

Sample 1:

Sample 2:

Sample 3:

(B.3) Calculate the mass % of acetic acid in vinegar, and then obtain the average value:

Sample 1:

Sample 2:

Sample 3:

AVERAGE:

**QUESTIONS**  
**Experiment 15**

1. How accurately does the 100 mL of water used to dissolve the KHP in the standardization of the NaOH solution need to be measured? Explain.
2. Why is it important to use the NaOH solution in the buret in Parts A and B of this experiment instead of putting vinegar in the buret in Part B? Explain.
3. If some KHP is lost upon transfer to the Erlenmeyer flask in Part A, how is the calculated molarity of the NaOH solution (based on the determined mass of KHP) affected compared to the actual value (what it should be due to less KHP)? Explain.
4. How does obtaining a dark pink endpoint (instead of a pale pink endpoint) in the titration in Part A affect the calculated molarity of the NaOH solution compared to the actual value (what it should be)? Explain.
5. How does obtaining a dark pink endpoint (instead of a pale pink endpoint) in the titration in Part B, affect the calculated mass % of acetic acid in vinegar compared to the actual value (what it should be)? Explain.

Name \_\_\_\_\_

Section \_\_\_\_\_

Date \_\_\_\_\_

**PRE-LAB EXERCISES**  
**Experiment 15**

INCLUDE THE APPROPRIATE SIGNIFICANT FIGURES.

1. Write the balanced molecular equation for the neutralization of acetic acid ( $\text{CH}_3\text{COOH}$ ) with sodium hydroxide.
2. Before the sodium hydroxide can be used in this experiment, it must be standardized (i.e. the concentration must be measured accurately). This lab will use  $\text{C}_6\text{H}_4(\text{COOH})\text{COOK}$  (KHP) to standardize the NaOH solution. Write the balanced molecular equation for the neutralization of KHP with sodium hydroxide.
3. Calculate the concentration of the sodium hydroxide solution (in M) if it takes 25.00 mL of sodium hydroxide solution to titrate 0.5000 g of KHP (MM = 204.22 g/mol).
4. Calculate the mass % of acetic acid in vinegar if you found 0.00400 mol of acetic acid in 5.000 mL of vinegar (density = 0.900 g/mL).

$$\text{Mass \% of component X} = \frac{\text{mass of X}}{\text{total mass of solution (containing X)}} \times 100$$