Experiment 15B

WBH 10-13-2022 FV

ANALYSIS OF VINEGAR

MATERIALS: 250 mL Erlenmeyer flasks (4), 50 mL Buret, Buret clamp, Buret funnel, 100 mL beakers (2), 5.00 mL volumetric pipet, pipet bulb, approx. 0.1 M NaOH, $C_6H_4(COOH)COOK$ (potassium hydrogen phthalate), phenolphthalein indicator, vinegar solution.

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. Define the meaning of the term "hygroscopic".
3. Describe the function and necessary qualities of a primary standard.
4. Perform a titration with precision and accuracy.
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_3COOH, is a weak monoprotic acid with a molar mass of 60.05 g/mole. In this lab, the percent by mass of acetic acid in vinegar will 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*. The titration analysis is based upon the acid-base neutralization reaction between acetic acid and sodium hydroxide solutions as shown below:

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

Prior to titrating an acid, it is first necessary to prepare the sodium hydroxide titrant solution. While it is a simple matter to weigh out some NaOH(s) and dissolve it in a known volume of water, it is *not* possible to accurately calculate the concentration of NaOH in the solution directly from a measured mass of NaOH. There are several reasons for this. First of all, sodium hydroxide is a *hygroscopic* solid, meaning that it readily absorbs water from the air. Thus, a given mass of NaOH(s) that has been exposed to the atmosphere contains an unknown mass of water. In addition to absorbing moisture, NaOH(s) also slowly absorbs atmospheric CO_2 to form some quantity of sodium hydrogen carbonate via the following reaction:

$$NaOH\ (s) + CO_2\ (g) \rightarrow NaHCO_3(s)$$

The same reaction also occurs in any NaOH solution that is exposed to the atmosphere, gradually decreasing its hydroxide concentration. While such reactions have important practical applications, such as scrubbing CO_2 from industrial gas streams, or emergency removal of CO_2 from the atmosphere inside a submarine or spacecraft, in this case it is just a nuisance.

In this experiment, the actual concentration of the sodium hydroxide solution will be determined (the solution will be "standardized") by using it to titrate a *primary standard* acid. A primary standard is a substance from which a solution of known concentration can be prepared. The primary standard acid used in this experiment is potassium hydrogen phthalate, $C_6H_4(COOH)COOK$ (204.22 g/mol), hereafter

abbreviated as "KHP". A monoprotic acid, KHP has several advantages: it can be obtained in very pure form, and dissolves readily in water; unlike NaOH, solid KHP is not hygroscopic, nor does it react with atmospheric CO_2; it is easily dried and weighed, and its relatively high molar mass makes it easy to weigh out samples containing small numbers of moles. The neutralization reaction between sodium hydroxide and KHP in aqueous solution is shown below:

$$\underset{}{NaOH(aq)} + \underset{204.22\ g/mol}{C_6H_4(COOH)COOK\ (aq)} \rightarrow C_6H_4(COONa)COOK\ (aq) + H_2O\ (l)$$

Because the reactants and products are all colorless, a visual acid-base indicator is used to determine when the acid-base neutralization reaction is complete. In this experiment, a small amount of the compound phenolphthalein added to the titration solution serves as the indicator. Phenolphthalein is colorless in the acidic KHP solution at the start of the titration, but appears pink in the basic solution formed when the KHP has been completely neutralized (at the equivalence point). Thus, the titration procedure involves slowly adding the standardized sodium hydroxide to the KHP solution until the appearance of a persistent faint pink color signals that the equivalence point has been reached.

PROCEDURE:

Note: Every workstation should have a plastic wash bottle filled with deionized water. You will be making regular use of this throughout the experiment. If necessary, refill the bottle from the white plastic spigot at the sink.

Setup. Preparation of Glassware and Buret.

1. Ensure that all glassware is clean. The Erlenmeyer flasks should be rinsed thoroughly with deionized water before use. It's OK if the inside of the flask is a little wet after rinsing (you're going to add more water anyway), however the **outside of the flask must be carefully dried** before weighing.

2. Rinse the buret with deionized water and drain through the stopcock into a waste beaker. Check that the stopcock is functional and does not leak. When you open the stopcock, do you observe a steady flow of liquid? If not, consult your instructor.

3. Using a clean, dry beaker, obtain some provided NaOH solution and carefully bring it to your hood (use this same beaker to obtain more NaOH and refill the buret as necessary throughout the experiment).

4. With the *stopcock closed*, pour several mL of the NaOH solution into the buret using the funnel provided. Remove the buret from the clamp, tip and roll it between your fingers to make sure the internal surfaces of the buret are completely rinsed with the NaOH solution. Then replace the buret in the clamp and open the stopcock to drain the rinsings into the waste beaker. Repeat this process a second time.

5. Once the buret has been thoroughly rinsed with the NaOH solution, ensure that the stopcock is closed, and then carefully fill the buret with NaOH using the funnel. Briefly open the stopcock "full bore" and drain a few mL of NaOH into the waste beaker. This operation should blow any bubbles out of the tip of the buret. The level of the NaOH in the buret should be at or below the 0.00 mL mark before starting the titration.

Part A. Standardization of the Sodium Hydroxide Solution

1. Rinse a 250 mL Erlenmeyer flask with deionized water. Make sure the outside of the flask is dry. Place the flask on an *analytical* balance and record the mass (to ±0.0001 g).

2. Place a small weighing boat on the *top-loading* balance, zero the balance, and then transfer about 0.50 g of KHP into the weighing boat. Dispose of any excess KHP in the waste beaker provided near the balance – do NOT return it to the bottle!

3. Carefully pour the KHP sample from the weighing boat into the pre-weighed Erlenmeyer flask. Then, using the *analytical balance*, record the mass of the Erlenmeyer flask + KHP (to ±0.0001 g)

4. 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.

5. Record the initial volume reading of the buret (to ±0.01 mL). Then carefully titrate the KHP solution with the NaOH until a pale pink endpoint is reached. The pink color should persist in solution for at least 30 seconds. Record the final volume reading of the buret. Calculate the mass KHP/mL NaOH ratio and enter into your data sheet.

6. Repeat steps 1-5 again. The mass KHP/mL NaOH ratios for the two samples should be within 2% of each other – if not, repeat the standardization titration again until you achieve two titrations with KHP/mL ratios that agree within 2%. ***NOTE: use ONLY the data for those two consistent runs in your calculation of the NaOH concentration***

Part B. Measurement of Acetic Acid in Vinegar

1. Use an analytical balance to obtain the mass of an empty 250 mL Erlenmeyer flask and record the value.

2. Pre-rinse a volumetric pipet with the vinegar solution. This rinse can be disposed of down the sink. Then, quantitatively measure 5.00 mL of the vinegar solution and place it in the pre-weighed 250 mL Erlenmeyer flask. Obtain the mass of the flask with the 5.00 mL of vinegar solution and record the value.

3. 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.

4. Refill the buret with NaOH as you did before part A. Record the initial volume.

5. Titrate the vinegar solution with the NaOH solution until a pale pink endpoint is reached. Record the final volume.

7. Repeat this process two more times. Each titration should be within 0.50 mL of each other.

Clean up:

1. All waste solutions from this experiment can be poured down the sink drain.

2. Rinse all glassware thoroughly with deionized water and arrange neatly in the hood. Clamp the rinsed buret on the ring stand with the tip pointing up and stopcock open to allow it to drain.

Name_____ Section_____

Partner_____ Date_____

DATA SECTION
Experiment 15B

INCLUDE THE APPROPRIATE SIGNIFICANT FIGURES.

Part A. Standardization of the Sodium Hydroxide Solution

	Sample 1	Sample 2	Sample 3 (if needed)
Mass of 250 mL Erlenmeyer flask (g)			
Mass of 250 mL Erlenmeyer + KHP (g)			
Mass of KHP (g)			
Initial buret reading (mL)			
Final buret reading (mL)			
Volume of NaOH used (mL)			
Value of (g KHP/mL NaOH) ratio			

NOTE: Need 2 good titrations with (mass KHP/mL NaOH) values within 2%

Part B. Measurement of Acetic Acid in Vinegar

	Sample 1	Sample 2	Sample 3
Mass of 250 mL Erlenmeyer flask (g)			
Mass of 250 mL Erlenmeyer + 5.00 mL Vinegar (g)			
Mass of 5.00 mL Vinegar (g)			
Initial buret reading (mL)			
Final buret reading (mL)			
Volume of NaOH used (mL)			

NOTE: Need 3 good titrations within +/- 0.50 mL

DATA TREATMENT
Experiment 15B

INCLUDE THE APPROPRIATE SIGNIFICANT FIGURES.

Part A. Standardization of the Sodium Hydroxide Solution

NOTE: ONLY include calculations for "good" samples having (mass KHP/mL NaOH) values within 2% of each other. At least two "good" samples are required for this part.

(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 15B

Part B. Measurement of Acetic Acid in Vinegar

NOTE: ONLY include calculations for "good" samples having titrant volumes within +/- 0.50 mL of each other. Three "good" samples are required for this part.

(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 15B

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. If some KHP is spilled on the benchtop during transfer from the weighing boat to the Erlenmeyer flask in Part A, how will 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.

3. 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.

4. 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.

PRE-LAB EXERCISES
Experiment 15B

Name _____ Section _____

Date _____

INCLUDE THE APPROPRIATE SIGNIFICANT FIGURES IN ALL CALCULATIONS

1. Write the balanced molecular equation for the neutralization of acetic acid (CH_3COOH) 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). In this lab, we will use the compound $C_6H_4(COOH)COOK$ (abbreviated "KHP") to standardize the NaOH solution. Write the balanced molecular equation for the neutralization of KHP with sodium hydroxide.

3. Suppose that a 0.5000 g sample of KHP (molar mass = 204.22 g/mol) is titrated with aqueous NaOH solution. If it requires 25.00 mL of sodium hydroxide solution to completely neutralize the KHP, what is the molarity of the NaOH titrant?

4. Calculate the mass % of acetic acid in vinegar if you found 0.00400 mol of acetic acid (molar mass = 60.05 g/mol) in 5.000 mL of vinegar (density = 1.01 g/mL).

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