

Experiment # 5  
VINEGAR: AN FDA INVESTIGATION

### Objective

In this experiment, you will play the role of an FDA analytical chemist, You will verify whether a vinegar manufacturer's quality control lab remains in compliance with the FDA regulations and standards.

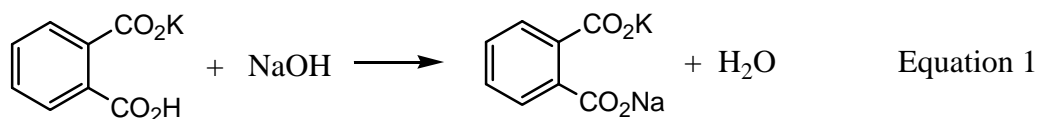
### Background Information

Regardless of the variety or brand name, vinegar is always an aqueous solution of an organic acid called acetic acid ( $M_m = 60.05 \text{ g/mol}$ ). Acetic acid is the active ingredient in vinegar. It is a chemical substance and as such is subject to analytical scrutiny. There are certain standards set by the FDA (Food and Drug Administration) for every food item that contains a chemical substance. According to those standards, the concentration of acetic acid in common vinegar cannot be less than 4 or more than 5 percent by weight. The acid content in vinegar is referred to as acidity. The acidity in each specific batch of vinegar produced is routinely verified by the quality control lab at the manufacturer's site.

The volumetric analytical technique called acid-base titration is commonly used to determine the acid or base content in a sample. In titration, a scientist precisely measures the volume of a solution (called a titrant) that completely reacts with a given mass or volume of a substance being analyzed (the analyte). The titrant may be a solution of known concentration with an unknown analyte, or the roles may be reversed. The titrant is added through an accurately calibrated buret (see below). The completion of the titration process, the end point, may be determined by the color change of a third substance that is added to the analyte before titration, in this case, a pH indicator. The end point, as signaled by the color change of the indicator, is achieved by adding just one additional drop of the titrant, after the equivalence point for the reaction has been reached.

### Standardization of NaOH

In order to accurately determine the concentration of acetic acid in vinegar, it is necessary to know the precise concentration of the titrant, sodium hydroxide. The molar concentration of NaOH is determined through a process called standardization. A standardized solution is one whose concentration has been accurately determined by titration against a precisely known amount of a primary standard. The primary standard must meet certain requirements: it must be a solid of known composition, must be extremely pure, be stable upon heating and exposure to air, and cannot be hygroscopic. In our experiment today, the primary standard will be an acid, potassium hydrogen phthalate or php (see equation 1).



The NaOH solution to be standardized is used to titrate a pre-weighed amount of the primary standard dissolved in water. Phenolphthalein, an indicator dye that turns pink above a pH of 8.3, is used to determine the endpoint. The number of moles of php ( $n_{\text{php}}$ ) is determined by multiplying the mass of php ( $m_{\text{php}}$ ) by its molar mass ( $M_m = 204.2 \text{ g/mol}$ ). The reaction equation (equation 1) indicates that there is a 1:1 molar ratio of php to NaOH. Therefore, the reaction

reaches its equivalence point when all moles of php present in the dissolved sample ( $n_{\text{php}}$ ) have been neutralized by an equivalent number of moles of NaOH ( $n_{\text{NaOH}}$ ). The delivered volume of the titrant ( $V_{\text{NaOH}}$ ) and the number of moles of NaOH ( $n_{\text{NaOH}} = n_{\text{php}}$ ) are then used to calculate the molar concentration of NaOH solution ( $M_{\text{NaOH}} = n_{\text{NaOH}}/V_{\text{NaOH}}$ ).

### Titration of vinegar

Once the NaOH solution is standardized, it can be used to determine the acidity of vinegar. Acetic acid present in vinegar reacts with NaOH according to the following equation:



Each mole of NaOH delivered from the buret reacts with one mole of acetic acid present in the vinegar sample tested. The reaction equivalence point is achieved after all moles of acid present in vinegar have reacted with the equivalent number of moles of NaOH present in the volume of titrant delivered from the buret. The end point is marked by the change of color of the phenolphthalein indicator from colorless to light pink (the pH at the end point is 8.3). Because the reaction occurs with a 1:1 molar ratio of reactants, the number of moles of acetic acid present in the vinegar sample is the same as the number of moles of NaOH delivered in the volume drained from the buret ( $n_{\text{acetic acid}} = n_{\text{NaOH}}$ ). Knowing the number of moles and the molar mass of acetic acid (60.0 g/mol), one can calculate the mass of acetic acid that is present in a 1.00 ml vinegar sample ( $m_{\text{acetic acid}}$ ).

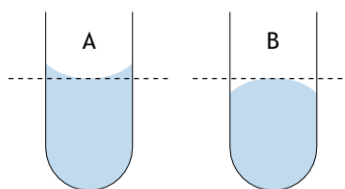
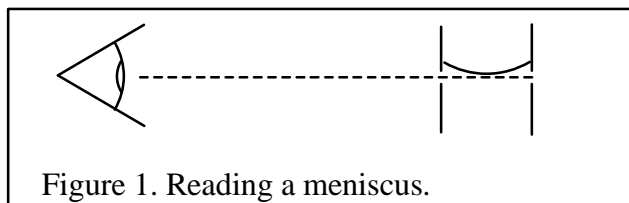
The mass of the vinegar sample must be calculated (the density of vinegar is 1.004 g/ml). The weight % of acetic acid in vinegar can then be calculated ( $m_{\text{acetic acid}}/m_{\text{vinegar}}$ ), as can the % error with respect to the acceptable FDA standard (equation 3).

$$\% \text{ Error} = \frac{\text{expected wt \%} - \text{measured wt \%}}{\text{expected wt \%}} \times 100\% \quad \text{Equation 3}$$

### Buret

A buret is a long tube, calibrated in ml and fitted with a stopcock that allows for precise control of titrant flow. The total capacity of the buret used in this experiment is 50.00 ml, with 0 marked on the top and the volume increasing down the buret to the mark of 50 ml. The smallest mark on the buret scale indicates 0.10 ml.

The buret is filled and the initial titrant volume read, at the bottom of the meniscus (Figure 1), and recorded. The meniscus is the curved upper surface of a column of liquid as it interacts with the walls of its vessel. The titration is performed by slowly adding the titrant, manipulating the stopcock with left hand while using right hand to swirl the flask containing the analyte solution. After the end-point has been reached, the final volume of the titrant is read and recorded. The volume of titrant used for the reaction is calculated by subtracting the initial volume from the final volume recorded.



- A. The bottom of a concave meniscus,
- B. The top of a convex meniscus

## Report

As an acting FDA agent, you are to prepare a formal, typed report. The report must include the following parts:

- **Objective** (goal):
- **Background:** write your own, do not plagiarize the text included above; it does not need to be extensive, but should definitely include a reference to the FDA standards, and methodology used.
- **List of equipment and reagents:**
- **Procedure:** you may reference the procedure in the text, but remember to attach the procedure. e.g.: "Refer to the enclosed procedure, *Vinegar, an FDA Investigation*"
- **Data:** you must retype the data in tabular format including all necessary modifications, but remember to attach the originally used data sheet.
- **Calculations:** write all involved math formulas using symbols (e.g.  $m_{\text{NaOH}}$  for mass of NaOH, etc.) not words, and include one set of corresponding calculations using numerical data. The calculations must be organized and labeled in such a way, that any educated person would understand the logic of every step. Remember that the report may be read by others who are not chemists. You must clearly document what was done and how, in order for others to verify the correctness of your report.

*Sample of a calculations plan (for part 1):*

1. Standardization of NaOH.

- a) number of moles of php:  $n_{\text{php}} = m_{\text{php}} \times (1/M_{\text{m php}})$ ; e.g.  $n_{\text{php}} = 0.11\text{g}(1.0 \text{ mol}/204.2 \text{ g})$
  - b) number of moles of NaOH:  $n_{\text{NaOH}} = n_{\text{php}}$
  - c) Molarity of NaOH:  $M_{\text{NaOH}} = n_{\text{NaOH}}/V_{\text{NaOH}}$
  - d) Average molarity of NaOH:  $M_{\text{NaOH}} = [M_1 + M_2 + M_3]/3$
- **Results and discussion of results:** state the final result and the calculated % error. Evaluate and discuss possible experimental errors (at least four) that could have affected the analysis. In other words, show the effect that a given error had on the final result including the step-wise logic, e.g. "the air bubble in the tip of a buret at the beginning of titration would seemingly increase the amount of titrant used, thus increasing the number of moles of NaOH calculated and, consequently, the number of moles of acetic acid reported. This would result in a calculation of higher acidity of vinegar because the number of moles is directly proportional to mass, and thus to mass percent". Without the error evaluation, no complete information is provided for an FDA official to be able to make an educated decision concerning the fate of the tested batch of vinegar.
  - **Conclusion:** a short statement concerning the manufacturer's compliance with FDA standards (quality of the specific brand and batch of vinegar) and a recommendation on any future action (should it stay on the store shelves?)

Remember that you are to write as if you are a serious FDA analyst. Do not include your feelings in the report, just the facts and their scientific implications. Use passive voice (e.g. ... The acidity of Best Yet vinegar, batch number 012345, was determined to be 4.5% ...)

## PROCEDURE

### I. Standardization of NaOH solution

#### 1. Preparation of the buret

- a. Obtain a ring stand and attach a buret clamp to it.
- b. Secure a buret in the clamp. The buret should be clean but always check to be sure.
- c. With the buret secured in a clamp and the stopcock closed, place a small funnel in the top opening. Obtain a clean and dry 50 mL beaker and fill it with the provided NaOH solution. Pour about 5 ml of the solution from the beaker into the buret.
- d. Take the buret off the stand and tilt it so that the walls come in contact with the NaOH solution.
- e. Place the buret in the clamp again and drain the rinse solution through the stopcock into a waste beaker. If the buret was wet to start with, repeat rinsing process with another 5 ml of NaOH.
- f. With the stopcock closed, fill the buret with NaOH a little above the 0 ml mark.
- g. Open the stopcock and let the titrant flow for a moment to fill the tip. To remove the air bubble, you may need to turn the stopcock back and forth in sharp, quick movements while tapping the tip with your fingers. Record the initial volume of titrant to 0.1 ml (it does not have to be 0.0). Remember to read the bottom of the meniscus.

#### 2. Standardization

- h. Using a spatula, transfer 0.10 g (within  $\pm 0.01$  g range) of potassium hydrogen phthalate (php) onto a folded weighing paper tared on the balance. Record the actual mass of php.
- i. Transfer the php sample to a 125 ml Erlenmeyer flask and add about 15 ml of distilled water and 2 drops of 0.1% phenolphthalein solution. Swirl the flask to dissolve the php. Not all of the php will dissolve at this point, but it will eventually, as titration progresses as long as you swirl the solution continuously.
- j. Position the flask with the php solution under the buret and raise the flask until half of the buret's tip is inserted into the flask. Place a sheet of white paper underneath the flask. Titrate, adding NaOH in small increments while swirling the flask. Slow the addition of the titrant when the pink color appearing after each addition dissipates at a slower rate (you may need to slow down to one drop per addition).  
The titration end-point is indicated by an extremely pale-pink color of the solution that persists for more than 15 seconds.

Repeat steps h-j two more times using a new sample of php and a clean flask each time.

### II. Analysis of vinegar

Refill the buret with NaOH as needed.

- k. Using a volumetric or Eppendorf pipet, transfer exactly 1.00 ml of vinegar into a clean 125 ml Erlenmeyer flask. Add about 15 ml of distilled water and 2 drops of phenolphthalein solution. Record the % acid in the vinegar sample as listed on the label.

1. Titrate with the standardized NaOH solution. Perform the titration three times using a clean flask and a new portion of vinegar each time.

**Buret cleanup**

Leave the buret secured to the stand. Drain the buret through the tip into a beaker. Dispose of the NaOH solution in the waste beaker provided. Close the stopcock, fill the buret with tap water and drain through the tip. Repeat one more time with tap water and then again with distilled water. Leave the stopcock open and return the buret to the instructor's cart.

**Chemistry 1225 experiment 5 PRELAB**

Name \_\_\_\_\_

1. In part 1, concerning the standardization of NaOH, the procedure asks that you weigh 0.10 g of php to within  $\pm 0.01$  g. This requirement constitutes the range of acceptable mass of KHP from \_\_\_\_\_ to \_\_\_\_\_ grams

2. An NaOH solution of unknown concentration was standardized using php. The table below contains the data collected for two titrations.

Table 1

	Determination 1	Determination 2
Mass of php (g)	0.20	0.22
Initial volume of NaOH in the buret (ml)	0.00	10.3
Final volume of NaOH in the buret (ml)	10.3	21.6

(A) Calculate the number of moles of php used?

(B) How many moles of NaOH were used?

(C) What is the molarity of the NaOH solution?

