# ANALYSIS OF COMMERCIAL VINEGAR

## Purpose

In this experiment you will prepare and standardize a NaOH solution by titration with a primary standard, KHP and determine the percentage of acetic acid in commercial vinegar by titration with the standardized NaOH solution. You will also measure pH and use it to classify the solutions.

## Introduction

This experiment is an introduction to volumetric analysis which is based on the measurements of volumes of reacting solutions. In acid-base and oxidation-reduction reactions, chemists use titration to determine the concentrations of solutions. The most commonly used concentration unit is molarity, M:

$$\mathbf{M} = \frac{moles}{L}$$

In this experiment, you will be performing acid-base titrations. The reaction that occurs in an acid-base titration is the neutralization reaction:

$$\mathbf{H^+ (aq) + OH^- (aq) \rightarrow H_2O (l)}$$

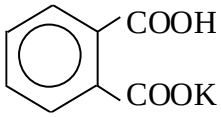
The $H^+$ ions are provided by the acid and the $OH^-$ ions from the base react with them.
In an acid-base titration, base is added from a buret to the acid in the titration flask until all of the acid has reacted. The completion of the reaction is termed the equivalence point. At the equivalence point of the titration, the number of moles of $H^+$ present has been neutralized by an equivalent number of moles of $OH^-$.

**At the equivalence point:** $\boldsymbol{n}_{H+} = \boldsymbol{n}_{OH-}$

It is necessary to know when the equivalence point has been reached. This is accomplished by using an indicator, which changes color when the reaction is complete. Phenolphthalein indicator is colorless in acid and red in base. When the base has reacted with all the acid, and no excess base has been added, the mixture will have a slight pink tint. This pink color signals the end of the titration.

In Part A of the procedure you will be standardizing a NaOH solution. Determining the concentration of a solution is known as standardization, and a standard solution is one whose concentration is accurately known. A primary standard is a chemical with a high degree of purity and stability. It is easily dried and doesn't absorb moisture from the air.

Potassium hydrogen phthalate is a primary standard, which has one acidic proton and has a molar mass of 204.2 g $mol^{-1}$.




Potassium hydrogen phthalate (KHP)

NaOH solutions have a tendency to absorb $CO_2$ from the air and as a result sodium carbonate and bicarbonate are formed, reducing the amount of free NaOH in solution.

$$CO_2 + H_2O \rightleftharpoons H_2CO_3$$

$$H_2CO_3 + NaOH \rightleftharpoons NaHCO_3 + H_2O$$

$$NaHCO_3 + NaOH \rightleftharpoons Na_2CO_3 + H_2O$$

Therefore NaOH solutions must be standardized before each use. KHP can be used to do this. In this experiment you will standardize a NaOH solution and then use this solution to determine the concentration of vinegar.

Example: Data: mass of KHP = 0.4251 g
volume of NaOH = 23.60 mL

Calculations:

At the equivalence point of the titration:

$$n_{NaOH} = n_{KHP}$$

$$c_{(\text{mol L}^{-1}\text{ of NaOH})} \times L_{(\text{NaOH})} = \frac{\text{mass}_{(\text{KHP})}}{\text{molar mass}_{(\text{KHP})}}$$

$$c_{(\text{NaOH})} = \frac{\text{mass}_{(\text{KHP})}}{\text{molar mass}_{(\text{KHP})} \times L_{(\text{NaOH})}}$$

$$c_{(NaOH)} = \frac{0.4251\text{g}}{204.2\,\text{g mol}^{-1} \times 0.02360\text{L}}$$
$$= 0.08821\,\text{mol L}^{-1}$$

Vinegar is a dilute solution (in water) of acetic acid, $CH_3COOH$. Acetic acid reacts with sodium hydroxide to produce sodium acetate and water. The chemical equation for the reaction is:

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

One mole of acetic acid reacts with one mole of sodium hydroxide, therefore at the equivalence point of the titration:

$$n_{\text{acetic acid}} = n_{\text{sodium hydroxide}}$$

Since

$$n_{\text{acid}} = n_{\text{base}}$$

And

$$n = c \times V$$

Then

$$c_{acid}V_{acid} = c_{base}V_{base}$$

The concentration of acetic acid in the dilute vinegar solution can be calculated using the formula above. To determine the concentration of the original vinegar solution, the dilution formula is used:

$$c_1V_1 = c_2V_2$$

Labels on vinegar bottles usually specify the percent acidity, which is defined as the number of grams of acetic acid per 100 mL of vinegar. The molar mass of acetic acid is 60.05 g $mol^{-1}$. Multiplying the concentration (mol $L^{-1}$) by 60.05 g $mol^{-1}$ will give the number of grams per liter and then dividing by 10 will give the number of grams per 100 mL. Using this information the percent acidity for the vinegar sample can be calculated.

**Example:** Given that $c_{vinegar} = 0.8172\ \frac{\text{mol acetic acid}}{\text{L vinegar}}$

$$\text{Percent acidity} = 0.8172 \ \frac{\text{mol}}{\text{L}} \times 60.05 \ \frac{\text{g}}{\text{mol}} \times \frac{1\ \text{L}}{1000\ \text{mL}} \times 100.00\ \text{mL}$$

$$= 4.907\ \%\ \text{(mass / vol)}$$

The solution has the composition 4.907 % mass by volume of $CH_3COOH$; that is, it contains 4.907 g $CH_3COOH$ per 100.00 mL of vinegar.

## pH

**All** aqueous solutions contain some hydrogen ions, if for no other reason than the dissociation of water. The pH scale is a convenient way of expressing hydrogen ion concentration in solutions.

pH is defined as:

$$\mathbf{pH = -\log [H^+]}$$

where $[H^+]$ is the concentration of hydrogen ions in the solution.

For pure water $[H^+] = [OH^-] = 1.0 \times 10^{-7}$ M and pH = 7 at 25 °C.

**In general, at 25 °C:**

In acidic solutions the $H^+$ concentration is in excess and the concentration will be higher than $1.0 \times 10^{-7}$ M, therefore the pH will be less than 7. The lower the pH, the more acidic is the solution.

In basic solutions the $OH^-$ concentration is in excess and the concentration of $H^+$ will be lower than $1.0 \times 10^{-7}$ M, therefore the pH will be greater than 7. The higher the pH, the more basic is the solution.

Acidic solutions: pH < 7.00

Basic solutions: pH > 7.00

Neutral solutions: pH = 7.00

## PROCEDURE: Part A

1. Weigh, on an analytical balance, between 0.4 and 0.5 grams of KHP. Record the exact mass (to 4 decimal places) on the data sheet. Dissolve the KHP in about 50 mL of distilled

water in an Erlenmeyer flask.

2. Add a few drops of phenolphthalein indicator to the flask.

3. Obtain approximately 150 mL of NaOH solution in a clean, dry 250 mL beaker.

4. Fill a clean buret with the NaOH solution. Use the remaining NaOH solution to refill the buret as necessary. **DO NOT** refill the buret after every titration.

5. Titrate the solution in the flask until it just turns pink and record the volume used. The pink color signals the end point of the titration. (The pink color should remain for at least 30 seconds. Record all buret volumes to 2 decimal places.)

6. Repeat the procedure once more.

7. Calculate the concentration of the NaOH solution for the two trials and then determine the average concentration of NaOH.

**PROCEDURE: Part B**

1. From the lab bench, obtain a 100 mL volumetric flask, a 10 mL volumetric pipet, and a 30 mL beaker containing about 20 ml of commercial vinegar.

2. Use the pipet to transfer 10.00 mL of the vinegar into the 100.00 mL volumetric flask.

3. Fill the flask to the calibration mark with distilled water, and invert several times to mix the contents.

4. Transfer this diluted vinegar solution to a clean, dry 150 mL beaker.

5. Rinse a 25.00 mL pipet with this diluted vinegar solution, and then pipet 25.00 mL of the solution into a 250 mL Erlenmeyer flask.

6. Add 3 - 4 drops of phenolphthalein to the solution, and titrate with the sodium hydroxide solution that you standardized in Part A. Record the buret reading in the data table and do the calculations to determine the concentration of the dilute vinegar solution, $\mathbf{c_{acid}}$.

**7.** Pipet another 25 mL of the dilute vinegar solution into another 250 mL Erlenmeyer flask and titrate this second sample to the phenolphthalein endpoint. **Keep this titrated solution for procedure C.**

**PROCEDURE: Part C**

1. Use the pH meter on the lab bench to measure the pH of the NaOH solution and record it in Data Table C.

2. Use the pH meter on the lab bench to measure the pH of the dilute vinegar solution (before titration) and record it in Data Table C.

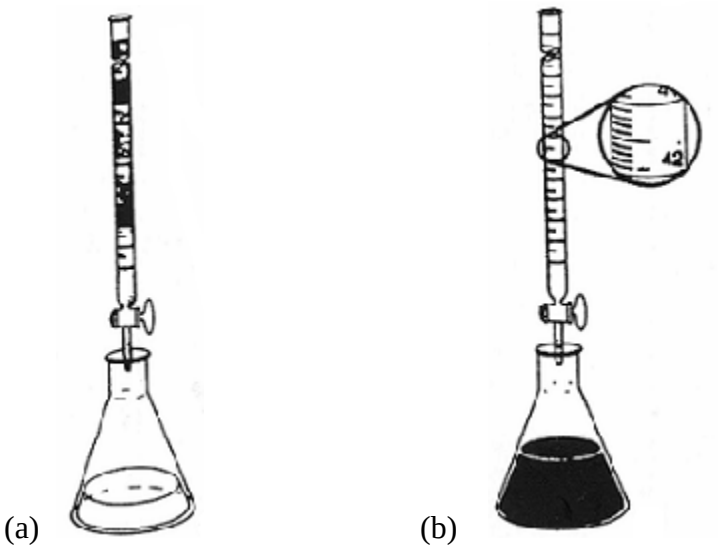
3. Use the pH meter on the lab bench to measure the pH of the titrated vinegar solution and record it in Data Table C.

4. Based on the pH measurement, classify each of the solutions as an acid, base, or neutral solution.

## TITRATIONS

Titration, or titrimetric analysis, is a volumetric procedure whereby a solution of accurately known concentration, called a standard solution, is added from a buret to a specific volume of another solution of unknown concentration, until the chemical reaction is complete. If we know the volumes of the standard and unknown solutions used in the titration, along with the concentration of the standard solution, we can calculate the concentration of the unknown solution.

The equivalence point of the titration is the point at which equivalent amounts of reactants have been mixed; both reactants have been consumed simultaneously and the reaction is complete. In order to determine the equivalence point, an indicator is added to the solution to be titrated. The indicator undergoes a sharp color change at (or very near) the equivalence point. The point of the titration where the indicator changes color is known as the **end point**. The closer it is to the equivalence point, the more accurate will be the analysis.



(a) (b)

**Figure 1 Titration.**
**(a) The unknown solution and indicator are placed in the flask.**

**(b) Solution is added from the buret to the flask. The end point is reached when the indicator changes color.**

At the end point the titration is stopped and the final buret reading is noted. Subtraction of the initial from the final buret reading gives the volume of standard solution that was added to reach the equivalence point of the titration. This information, together with the knowledge of the reaction stoichiometry – obtained from the balanced equation for the reaction – allows the analyst to calculate the concentration of the second solution.

**BURETS**

A buret is designed to deliver a precisely measured volume of liquid. It consists of a narrow graduated tube equipped with a stopcock for controlling the flow of liquid. Typically, 50 mL burets are used in chemistry labs.

**<u>Cleaning the Buret</u>:**

Like all volumetric glassware, a buret must be clean and grease free so its inner surface will drain evenly. Rinse the buret several times with tap water. Allow the water to drain out.

a) If a continuous film of water forms, wetting the inner surface uniformly, the buret is clean. Follow with three rinsings (small portions) of distilled water, allowing some of each portion to drain through the tip.

b) If individual water droplets form on the inside surface, the buret is greasy and should be washed with soap and tap water, using a buret brush. Next, rinse the buret thoroughly with tap water to remove any soap film, and then rinse with small portions of distilled water, allowing some of each portion to drain through the tip.

Because of their long narrow tube construction, burets are difficult to dry. Therefore, do not try to dry the burets!

**Filling the Buret:**

Pour the solution with which you will be filling the buret into a *clean, dry beaker*. Rinse the buret three times with approximately 10 mL portions of the solution it is to contain, allowing some of the solution to flow out through the tip each time. When rinsing, hold the buret almost horizontally and roll the buret so that the entire surface inside the buret is coated by the solution. Discard the rinsings.

To fill the buret, first make sure the stopcock is closed, then hold the buret in your hand at chest or waist level and tilt the buret. Pour the solution slowly and carefully from the beaker into the buret. Fill the buret up past the zero mL mark and then drain some solution through the tip. Ensure that the solution is free of air bubbles. Air bubbles above the stopcock can usually be removed by gently tapping with your finger. To displace air from the buret tip, drain a few mLs of solution through it.

*Avoid filling the buret when it is clamped in a vertical position*. When you reach up over your head to pour solution into the buret, drops of solution could easily run down your arm. Also, air bubbles are easily incorporated into the solution, and you want to measure the volume of solution, not the volume of air.

**Reading the Buret:**

After filling the buret, place the buret in the buret clamp. Make sure the buret is in the vertical position (not tilted in the clamp). Adjust the initial volume by draining some solution through the tip. It isn't necessary to adjust the starting volume to precisely 0.00 mL.

A 50 mL buret has calibrations divided into 0.1 mL intervals. Volumes can be estimated to the nearest 0.01 mL by mentally dividing each interval into 10 parts.
Each buret reading should have two digits after the decimal point. When reading the buret

volume, make sure your eye is level with the liquid surface and read across the bottom of the meniscus.

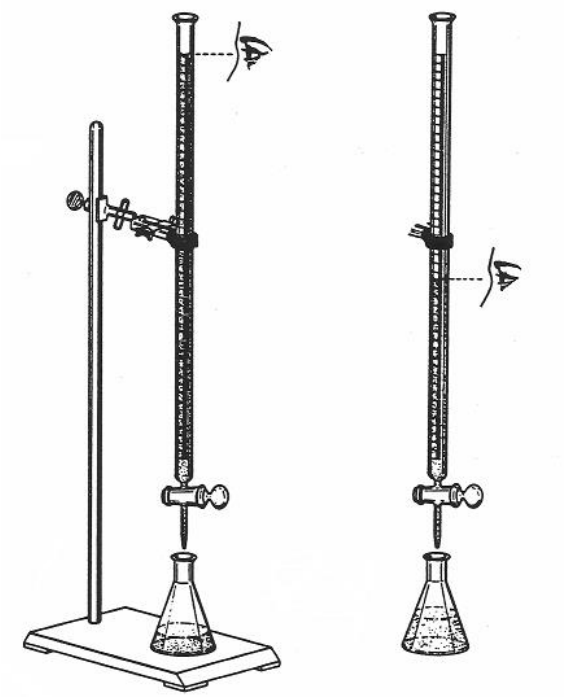


**Figure 2 Reading the buret**

**Titration Technique:**

The buret tip should extend 2 to 3 cm into the titration flask. The flask should sit on a white background so color changes are most easily seen. As you add solution from the buret to the flask, manipulate the stopcock with your left hand and swirl the flask with your right. Introduce the titrant in increments of about 1 mL. Swirl constantly to ensure thorough mixing. When you are within 1 or 2 mL of the end point, stop adding titrant and rinse down the inner walls of the flask with a minimum amount of distilled water to wash spattered droplets down into the solution. Now add titrant at a slow, drop-wise speed. When you think you are within a couple of drops of the end point, touch off the droplet on the tip and rinse down the flask again. Add one more drop, touch off the droplet on the tip and rinse down the wall of the flask at the touching-off point. Repeat this procedure until the end point color change occurs. Careful workers add half-drops instead of full drops at this stage. Wait about 15 seconds before taking a buret reading; this allows time for the liquid to drain down the inner walls of the buret. When titrating you must be careful never to allow the liquid level in the buret to fall below the 50 mL mark. If you do, the second measurement cannot be made -guessing isn't accurate- and you will have to

start over.

If you find that reaching the end point of the titration will require more solution than your buret currently contains, continue dispensing to *almost* the 50 mL mark, record the final volume measurement and calculate how much titrant you have added. Then refill the buret, record the new initial reading and continue titrating until the end point is reached. Record the final volume measurement and determine the total volume of titrant needed to reach the end point.

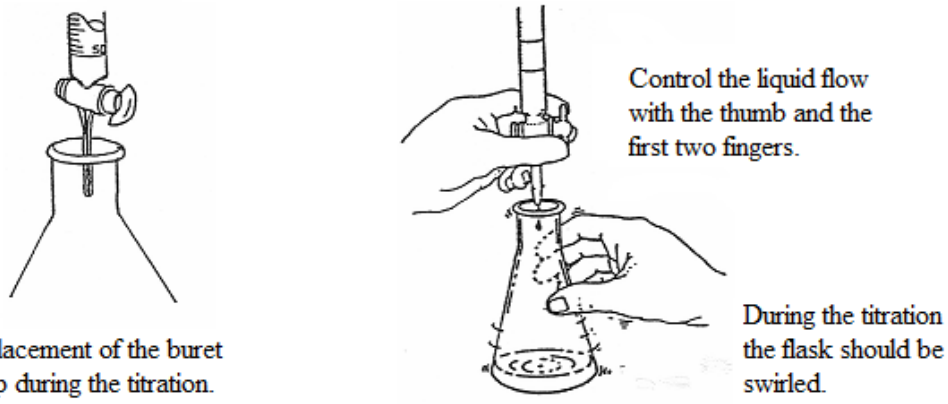



**Figure 3 Recommended method for manipulation of the buret stopcock.**

The volume of liquid dispensed from the buret into the receiving flask is calculated by subtracting the initial volume reading from the final volume reading:

| | |
|---|---|
| Final volume | 18.94 mL |
| Initial volume | 0.15 mL |
| Volume of titrant added | 18.79 mL |

**Clean-up:**

Rinse out the buret well with tap water, including the stopcock and tip. Give it a few final rinses with small portions of distilled water.