INTRODUCTION

Vinegar is prepared commercially in two steps, both requiring microorganisms. The first step is the production of ethyl alcohol, \( \text{C}_2\text{H}_5\text{OH} \), from sugars in grapes, apples, etc by yeast.

\[
\text{C}_6\text{H}_{12}\text{O}_6 \rightarrow 2 \text{C}_2\text{H}_5\text{OH} + 2 \text{CO}_2
\]

The second step is the conversion of ethyl alcohol to acetic acid by bacteria as illustrated by the following process:

\[
\text{C}_2\text{H}_5\text{OH} + \text{O}_2 \rightarrow \text{HC}_2\text{H}_3\text{O}_2 + \text{H}_2\text{O}
\]

The reaction of acetic acid with the base sodium hydroxide is:

\[
\text{HC}_2\text{H}_3\text{O}_2 + \text{NaOH} \rightarrow \text{NaC}_2\text{H}_3\text{O}_2 + \text{H}_2\text{O}
\]

The process of determining the amount of constituent in a sample by measuring the volume of a standard solution of known concentration that reacts with it in some definite and complete manner is known as titration. In the titration of an acid with a base, the point at which the moles of acid are equivalent to the moles of base is called the equivalence point. In the titration of vinegar, all components are colorless so a small amount of a substance called an indicator is added. An indicator is one color in the presence of excess acid and a different color in the presence of excess base. In this experiment phenolphthalein is used as the indicator. It will be colorless in the vinegar solution and turn a pink color when all the acetic acid is neutralized and the solution turns basic. The point in the titration at which the indicator turns color is called the end point. It does not exactly correspond to the equivalence point, but the discrepancy is usually insignificant.

The accuracy of a titration is limited in part by the accuracy with which the concentration of the standard solution is known. The concentration of a standard solution is established in one of two ways:

1. Careful preparation by accurately weighing a pure reagent and carefully diluting it to an exactly known volume.

2. Titrating the standard solution with a known mass of a pure compound using a reaction of known stoichiometry.
Lab # 7: Molarity of Acetic Acid in Vinegar: A Titration Experiment

You will be using the second method to standardize the NaOH solution to be used in the vinegar titration. The pure compound used for standardization is called a primary standard. Some important requirements for a primary standard are that it must:

1. be of the highest purity.
2. have chemical stability. It should not react with the atmosphere and should be nonhygroscopic (will not absorb water from the air).
3. have a high gram formula weight to minimize weighing errors.
4. react in a known manner with the reagent to be standardized.

A common primary standard for the standardization of bases is acid potassium phthalate (or potassium hydrogen phthalate), $\text{HKC}_8\text{H}_4\text{O}_4$, usually abbreviated KHP where the $P$ stands for phthalate, not phosphorus. It has a molecular weight (gram formula weight) of $204.2234 \text{ g/mol}$. It reacts with sodium hydroxide according to the following equation:

$$\text{HKC}_8\text{H}_4\text{O}_4 + \text{NaOH} \rightarrow \text{NaKC}_8\text{H}_4\text{O}_4 + \text{H}_2\text{O}$$

or

$$\text{KHP} + \text{NaOH} \rightarrow \text{NaKP} + \text{H}_2\text{O}$$

In this experiment you will prepare approximately 400 mL of a NaOH solution and determine its exact molarity by titration with KHP. The standardized NaOH solution will then be used to determine the molarity of acetic acid in an unknown acetic acid sample.

PROCEDURE

PREPARATION OF THE NaOH SOLUTION

Add approximately 400 mL of distilled water to a Florence flask. Then add about 15 mL of 6 M NaOH to the water, rinsing the graduated cylinder with a couple of milliliters of distilled water. Add the rinsings to the flask and MIX WELL. (To facilitate mixing, you may pour the solution back and forth several times between the flask and your largest, clean but not necessarily dry beaker. Look carefully at the solution; if you see “swirls” of inhomogeneity, your solution is not mixed well.) You will use this solution for all the titrations today. Do NOT discard it until the very end of the experiment.
STANDARDIZATION OF THE NaOH SOLUTION

Obtain a 50 mL buret and check for leaks. Rinse the buret twice with tap water and once with distilled water. (If the buret is really dirty, wash first with soap and water.) Then rinse the buret twice with 5 mL portions of your dilute NaOH solution, discarding the wash solution. Fill the buret with the NaOH solution, making sure that the tip of the buret is full and free from air bubbles. Record the initial volume on the buret to the nearest 0.01 mL.

Using weighing paper or boats, weigh four samples of between 0.7 and 0.8 g of KHP into four separate, clean Erlenmeyer flasks (125 or 250 mL). Make sure that all the KHP ends up in the appropriate flask. Add approximately 50 mL of distilled water to each of the flasks and swirl to dissolve the KHP. It may not completely dissolve at this point but will when you add NaOH during the titration. Add 2 or 3 drops of phenolphthalein indicator to each flask.

Starting with the flask containing the smallest amount of KHP, titrate each KHP solution with the NaOH, with constant swirling and frequent (but not excessive) rinsing, until all the KHP is dissolved and a stable pale pink color persists throughout the mixture for at least 30 seconds. Record the final volume of NaOH. You should obtain 3 good titrations from which to calculate the average molarity of your sodium hydroxide.

TITRATION OF THE ACETIC ACID SAMPLE

Obtain an unknown vinegar sample and record its number. Obtain a 5.00 mL volumetric pipet, rinse it twice with tap water, once with distilled water and twice with small portions of your unknown. Using the pipet, carefully and accurately measure 5.00 mL of your unknown acid sample into four clean (but not necessarily dry) Erlenmeyer flasks. Add 2 or 3 drops of phenolphthalein indicator and 50 mL of distilled water to each flask. Titrate each solution with the NaOH solution until you reach the pale pink endpoint. You should obtain 3 good titrations from which to calculate the average molarity of your acetic acid sample.

WASTE - All the waste from this experiment can be rinsed down the drain with water.
**DATA AND CALCULATIONS**

**STANDARDIZATION OF NaOH**

Check the 3 (at least) trials used to calculate the average molarity.

<table>
<thead>
<tr>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
<th>Trial 4</th>
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<tbody>
<tr>
<td>Mass of KHP</td>
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<td>Moles of KHP</td>
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<td>Initial buret reading</td>
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<td>Final buret reading</td>
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<td>Volume of NaOH used</td>
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<td>Moles of NaOH used</td>
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<td>Molarity of NaOH</td>
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Average molarity of NaOH ________________

**TITRATION OF ACETIC ACID SAMPLE – UNKNOWN NUMBER _____________**

Check the 3 (at least) trials used to calculate the average molarity.

<table>
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<tr>
<th>Trial 1</th>
<th>Trial 2</th>
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<td>Volume of NaOH used</td>
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<td>Molarity of acetic acid</td>
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Average molarity of acetic acid sample ________________

Instructor’s Initials _____________

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PRESTUDY

1. (5) A 0.7483 gram sample of KHP (204.2234 g/mol) requires 14.98 mL of a NaOH solution to completely neutralize it using phenolphthalein as an indicator. What is the molarity of the NaOH solution?

2. (5) A 10.00 mL sample of an acetic acid solution requires 35.87 mL of a 0.2174 M NaOH solution to completely neutralize it using a phenolphthalein indicator. Calculate the molarity of the acetic acid solution.