I. Introduction

Household vinegar’s unique smell is due to the presence of acetic acid in the solution. Manufacturers report the concentration of acetic acid on the bottle’s label as a mass percent. Typical acetic acid concentrations are in the 4% - 8% range. The USDA has required manufacturers to maintain a minimum organic acid percentage no lower than 4% in household vinegar.

Before titrating a vinegar sample you will perform a standardization procedure that accurately determines the concentration of the NaOH solution that is later used to titrate the vinegar. By reacting NaOH with a known amount of the organic acid potassium hydrogen phthalate (a.k.a. KHP or $\text{KHC}_8\text{H}_4\text{O}_4$), we will be able to determine the concentration of NaOH. Because the KHP is a solid that is weighed out on an analytical balance, it is possible to determine the number of grams and moles of KHP with 4 significant digits. Consequently, the concentration of NaOH can be determined with four significant figure accuracy also.

**IMPORTANT NOTE:** KHP doesn’t stand for potassium (K) hydrogen (H) phosphorus (KHP). To determine the molar mass of KHP you must use the following formula: $\text{KHC}_8\text{H}_4\text{O}_4$

The indicator used in all titrations is phenolphthalein. As NaOH is added to the vinegar or KHP solutions, the base is neutralized by the acetic acid. When all of the acid is used up, a slight excess of OH$^-$ ions develops. These ions react with the phenolphthalein molecule (Figures at right) producing the pink colored species that signals the endpoint of the titration.

When the titration is performed correctly, only a slight excess of hydroxide ions (OH$^-$) is present when the endpoint is reached signified by a light pink color. The equivalence point occurs before the endpoint and is when the NaOH has perfectly neutralized the KHP with no excess OH$^-$. 

II. Hazards 0.1 M NaOH

**WARNING! HARMFUL IF SWALLOWED. MAY CAUSE IRRITATION TO SKIN, EYES, RESPIRATORY TRACT AND GASTROINTESTINAL TRACT.**

Health Rating: 2 - Moderate  
Flammability Rating: 0 - None  
Reactivity Rating: 1 - Slight  
Contact Rating: 2 - Moderate  
Lab Protective Equip: GOGGLES; LAB COAT; VENT HOOD; PROPER GLOVES

The health effects from exposure to diluted forms of this chemical are not well documented. They are expected to be less severe than those for concentrated forms which are referenced in the descriptions below.

Inhalation: Mists are irritants to respiratory tract.  
Ingestion: Corrosive. Swallowing may cause burns of the mouth, throat and stomach.  
Skin Contact: Can be corrosive to skin. May cause irritation.  
Eye Contact: Sodium Hydroxide: Corrosive! May cause irritation of eyes, and with greater exposures, severe burns with possibly blindness resulting.  
Chronic Exposure: Prolonged contact can dehydrate and remove oils from skin.  
Aggravation of Pre-existing Conditions: Persons with pre-existing skin disorders may be susceptible to these solutions.
III. Experimental Procedure

Your objective in this lab is to first accurately determine the concentration of the provided NaOH solution. Then, you will use the NaOH solution's concentration to determine the concentration of acetic acid in a vinegar solution.

Suggestions:
- Today, you will work with a partner. Work efficiently and you will finish on time.
- You will submit individual lab reports.
- Good lab technique is important when performing a titration. Be sure you download and read the following materials before coming to class:
  - Volumetric Pipette Operation Guide
  - Volumetric Flask Operation Guide
  - Buret Operation Guide
  - Buret Demonstration (VIEW BEFORE COMING TO CLASS)

A. Equipment and Materials

- Small Plastic Funnel: used to fill the volumetric flask
- 250 mL beaker: temporary storage for waste solutions
- (2) 100 mL beakers: DRY! One for each KHP titration
- 50 mL beaker: undiluted vinegar reservoir
- 50 mL beaker: contains the vinegar solution to be titrated
- Buret: contains the NaOH solution dispensed into beaker above
- 250 mL volumetric flask: used in the preparation of vinegar solution
- 20 mL volumetric pipette: used in the preparation of vinegar solution
- Pipette helper: suction bulb that is used with vol. pipette
- Stir bar: used with the stir plate to keep solutions stirred during titration
- Medicine dropper: used to adjust the volumetric flask liquid level
- Analytical balance: used to weigh out KHP to 4 decimal digits
- Top loading balance: used for initial KHP weighing
- Phenolphthalein: acid base indicator
- 0.1 M NaOH: used to titrate KHP and vinegar
- KHP(s): Used to standardize 0.1 M NaOH solution
- Store-bought vinegar

B. Standardization of NaOH

The goal of this procedure is to accurately determine the concentration of the NaOH. This concentration will be used to determine the concentration of acetic acid in vinegar in part C.

1. Obtain and determine the mass of a clean 100 mL beaker using the analytical balance.
2. Weigh out approximately 0.3 grams of KHP (KHC\textsubscript{8}H\textsubscript{4}O\textsubscript{4}) into the 100 mL beaker using a top loading balance (i.e. pre-weigh the KHP).
3. Reweigh the beaker and KHP on the analytical balance and determine the amount of KHP to the nearest 0.1 mg (i.e. 4 decimal digits)
4. Using the markings on the side of the beaker as a guide, add approximately 30 mL of additional distilled water to KHP.

Prelab Question: Why is the exact amount of water not important?
5. Place a small magnetic stir bar in the solution and use the stir plate function of the hot plate (NO HEAT!!!) to stir the solution until all of the KHP has dissolved.

Avoid stirring so fast that the solution is splashed up on to the sides of the beaker.

Rinse any solution that splashes up on the sides of the beaker with a small amount of distilled water from a squirt bottle.

6. Add 3 drops phenolphthalein indicator to the solution in the beaker.

7. Obtain a 25 mL buret and rinse it twice with small amounts (1-2 mL) of the NaOH solution.

8. Fill the buret with NaOH solution and then drain a small amount of NaOH from the buret to remove bubbles trapped in the tip.

Remove the funnel from the top of the buret and record in your the initial reading (2 decimal digits) on your data sheet.

9. Use your buret to slowly add NaOH small amounts of NaOH solution to the 100 mL beaker. Stir at all times.

Initially, look for a pink color near where the NaOH enters the bulk solution. If you fail to see pink you have forgotten to add the phenolphthalein solution and must do so before continuing.

10. As the endpoint is reached, the color change will persist for longer times. Reduce the amount of NaOH you add as your approach the endpoint.

**Note:** During a titration, the meniscus must always fall on the buret's graduated scale! Don't let the meniscus drop below the 25 mL mark or you will have to start over again!

11. Accurately determine the endpoint of your titration using the hanging drop technique described in the buret operational guide.

12. When the pink color persists for 30 seconds or more, record the final buret reading with 2 decimal places on your data sheet. Calculate the amount of NaOH dispensed by subtracting the before and after readings.

13. Determine the number of moles of KHP \((\text{KHC}_8\text{H}_4\text{O}_4)\) using its molar mass and use this result to calculate the concentration of NaOH (4 sig figs) for the trial. The balanced chemical equation describing this reaction is:

\[
\text{KHC}_8\text{H}_4\text{O}_4(\text{aq}) + \text{NaOH}(\text{aq}) \rightarrow \text{KNaC}_8\text{H}_4\text{O}_4(\text{aq}) + \text{H}_2\text{O}(l)
\]

14. Repeat the above steps for a second KHP sample. If you don't obtain comparable results for the NaOH concentration, repeat a third time

**Calculating NaOH concentrations should be within +/- 0.0040 M.**

15. Calculate the average NaOH for these trials and record it on the data sheet (use excess S.F.).
C. Diluting the Vinegar

Standard vinegar is too concentrated to titrate using the \(~0.1\ M\ NaOH\) available in this experiment. Therefore, it is necessary to carefully dilute the vinegar with distilled water before titrating.

1. Obtain a 250 mL volumetric flask, 20 mL volumetric pipette and a pipette helper.
2. Pour approx 30 mL of white vinegar into a clean/dry 50 mL beaker.
3. Fill the volumetric pipette with vinegar to the fill mark and dispense the liquid into the 250 mL volumetric flask using the correct technique.
   
   Be sure to transfer the drop that adheres to the tip of the pipette to the volumetric flask by touching the flask's inside surface with the pipette's tip. The small amount of solution that remains inside the pipette must **NOT** be added to the flask.
4. Add distilled water to the 250 mL volumetric flask until the level of the liquid is near the measurement mark on the neck of the flask then continue to add distilled water with a medicine dropper until the liquid's meniscus and the fill mark are properly lined up. (Keep fill mark at eye level!) As you fill, take careful aim to avoid droplets clinging to the sides of the volumetric flask's neck that will affect the accuracy of the dilution.
5. Stopper the flask, **hold the stopper securely** and invert (turn upside-down) the volumetric flask at least 20 times to thoroughly mix the vinegar and water.

This solution will be used in part D.

D. Titration of Vinegar

A 20.00 mL sample of the diluted vinegar from part C is titrated with the NaOH solution standardized in part A. From these results the concentration of acetic acid in Vinegar is determined.

1. Rinse your 20mL pipette twice with the dilute vinegar solution you prepared in part C. Dispose of your rinse solutions in a waste beaker.
2. Pipette 20.00 mL of the solution prepared in part C into a clean 50mL beaker and add 3 drops of phenolphthalein to the beaker.
3. Rough Titration #1. Your first vinegar titration is performed crudely and quickly to determine the approximate endpoint of your titration.
   
   Fill your buret with the NaOH solution you standardized in part B (don't take from a different bottle!). Record the initial NaOH liquid level position on your data table. Add a small magnetic stir bar and add NaOH in 1 mL increments until the endpoint is reached. Record this value.
4. Careful Titration #2. Use your knowledge of the titration's endpoint location from step 3 and repeat the vinegar titration CAREFULLY. Quickly add NaOH until you are within 2 mL of the endpoint and then proceed carefully using small increments until a light pink color is obtained. Calculate and record the volume of NaOH used for each of these two trials.
5. Calculate the molarity of the acetic acid in vinegar. The balanced chemical equation is

   \[
   \text{CH}_3\text{COOH} + \text{NaOH} \rightleftharpoons \text{CH}_3\text{COONa}_{(aq)} + \text{H}_2\text{O}_{(l)}
   \]

6. Careful Titration #3. Repeat the above procedure for Careful titrations a second time using another 20.0 mL portion of the dilute vinegar solution found in the volumetric flask you prepared earlier in part C.
Your individual experimental report will be due at the beginning of class next week.

Data Table: (All entries must be in written in ink before you leave the lab).

### Part B: NaOH Standardization

<table>
<thead>
<tr>
<th></th>
<th>KHP Trial 1</th>
<th>KHP Trial 2</th>
<th>KHP Trial 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass: Beaker</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mass: Beaker + KHP</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Mass: KHP</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Molar Mass: KHP</td>
<td>(g/mol)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Moles: KHP</td>
<td>(mol)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial Buret: NaOH</td>
<td>(mL)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Final Buret: NaOH</td>
<td>(mL)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume: NaOH</td>
<td>(mL)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Moles: NaOH</td>
<td>(mol)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Concentration: NaOH</td>
<td>(M)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Average Concentration: NaOH (Report with excess S.F.)

### Part D: Vinegar Titration Results

<table>
<thead>
<tr>
<th>Brand Name of Vinegar</th>
<th>Vinegar Trial 1 (Rough)</th>
<th>Vinegar Trial 2 (Carefully)</th>
<th>Vinegar Trial 3 (Carefully)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Average Concentration: NaOH</strong></td>
<td>(mol/L)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial Buret: NaOH</td>
<td>(mL)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Final Buret: NaOH</td>
<td>(mL)</td>
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<tr>
<td>Volume: NaOH</td>
<td>(mL)</td>
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</tr>
<tr>
<td>Moles: NaOH</td>
<td>(mol)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Moles: CH₃COOH (dilute 20 mL sample)</td>
<td>(mol)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Moles: CH₃COOH (dilute 250 mL vol. flask)</td>
<td>(mol)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Moles: CH₃COOH (original 20 mL vinegar)</td>
<td>(mol)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>* Conc. : CH₃COOH (original 20 mL vinegar)</td>
<td>(M)</td>
<td></td>
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</tr>
</tbody>
</table>
Answer the following questions.

- Answers must be readable and make sense for full credit.
- Copied answers will result in all involved students receiving a zero score

1. For your first NaOH standardization trial, show all of the calculations you used to determine the NaOH concentration.

2. How do the number of moles of CH₃COOH in the original 20 mL vinegar sample compare to the number of moles of CH₃COOH in the 250 mL volumetric flask just after dilution? Why?

3. Calculate the molar concentration of CH₃COOH in the original 20mL undiluted vinegar sample. (Refer to * in the data table. Value should be ~0.8XXX Molar. Show one calculation but report both Trial 2 and 3 molarities.

4. Convert both molar concentrations from question 3 into mass percent. (Density vinegar = 1.005 g/mL).

   Hint: Convert... Molarity = mol CH₃COOH / L soln into Mass % = g CH₃COOH / g solution x 100%

   Report both Trial 2 and 3 results but show one sample calculation.

5. Calculate the average mass % of acetic acid using both vinegar trials from question 4.