Experiment 69

Analysis of Vinegar

Problem

How can the acid content of vinegar be determined experimentally?

Introduction

Ordinary "white" vinegar is an aqueous (water) solution of acetic acid which often carries the notation that the acidity has been reduced to 5% with water. Flavored products, like apple cider vinegar, red wine vinegar and balsamic vinegar have other ingredients and flavorings, but even they are essentially acetic acid in water.

In order to ensure that the acidity is at the desired level, periodic routine analyses are run. A common method for such analyses is a titration, in which a strong base of known concentration is used to determine the concentration of the acid by allowing the solutes of the two solutions to react with each other. In a titration, a solution (called the titrant) is added at a controlled rate to a known amount of the solution to be analyzed. Addition continues until the reaction is complete.

An indicator is often used to determine when all of the solute of the solution being tested has reacted. Indicators signal that a reaction is complete by changing color. Phenolphthalein, the indicator that you will use in this experiment, is colorless in acidic or neutral solutions, but turns bright magenta with the slightest excess of base. The first drop of base that causes the color to persist signals the end of the titration. The equation for the reaction between the sodium hydroxide and the acetic acid in the vinegar is:

\[ \text{HC}_2\text{H}_3\text{O}_2\ (aq) + \text{NaOH}\ (aq) \rightarrow \text{Na C}_2\text{H}_3\text{O}_2\ (aq) + \text{H}_2\text{O}\ (l) \]

The hydroxide ion of the base (NaOH) reacts with a hydrogen ion from the acid (HC\textsubscript{2}H\textsubscript{3}O\textsubscript{2}) to form water. These reactions are called neutralization reactions, because the acid and the base neutralize each other, producing water. Notice that only one hydrogen atom on the acetic acid molecule reacts with OH\textsuperscript{-}. Many common organic acids contain some hydrogen atoms that are acidic and others that are not. The difference need not concern you here; it is enough to know that acidic hydrogens will react with a base, but the others do not.

You will prepare a sodium hydroxide solution of known concentration, then use that solution to analyze the acid content of white vinegar. Once you determine the molar concentration (molality) of the sodium hydroxide, you will convert the concentration to units of moles of NaOH per gram of solution.

Because in the experiment both the mass and the concentration of the sodium hydroxide titrant are known, the number of moles of NaOH that reacts can be calculated. As the equation shows, acetic acid and sodium hydroxide react in a 1:1 mol ratio, so you can also
determine the number of moles of acetic acid present in the sample which can then be converted to mass in grams.

You will carry out four trials for the analysis. The amounts of the two solutions used in each of the titrations will be determined by weighing the pipettes before and after each of the titrations.

Prelaboratory Assignment

- Read the Introduction and Procedure before you begin.
- Answer the Prelaboratory Questions.

1. Write the equation for the reaction between acetic acid and NaOH. Use the structural formula for acetic acid found in Chapter 16. In the formulas of the reactants, circle the atoms that form water.

2. What is the purpose of the indicator? How does it tell you when a titration is complete?

3. Give two reasons for Safety Special Note #2. (Hint: Consider both your own experiment and that of the person who uses the balance after you.)

4. What does the parenthetical expression (± 0.001 g) mean in Step 1 of Part 1 of the Procedure?

5. Read Step 4 of Part 1 of the Procedure. Answer the question that appears in parentheses.

Materials

Apparatus

- Milligram balance
- Beaker, 30- or 50-mL
- Graduated cylinder, 25-mL
- 4 Erlenmeyer flasks, 10-mL (or small beakers)
- 2 microtip pipets, labeled NaOH and Vinegar
- Distilled water wash bottle
- Safety goggles
- Lab apron

Reagents

- Sodium hydroxide, solid pellets
- Phenolphthalein indicator, in microtip pipet
- 15 mL vinegar (includes some for Cleaning Up)

Safety
Analysis of Vinegar

1. Laboratory goggles and a lab apron must be worn at all times in the laboratory.

2. Sodium hydroxide is highly caustic (see Special Notes below). Avoid contact with skin and clothing and wipe up all spills.

Special Notes:

1. Working With Solid NaOH: The pellets are highly caustic. They will harm skin and clothing, and they will attack metal surfaces such as the pan of the balance. In addition, they rapidly absorb moisture from the air, so it is critical that Part A of the procedure be done quickly and efficiently.

2. Weighing Glassware: Always dry the outside surfaces of beakers, flasks, and other pieces of glassware before you place them on the balance pan.

3. Weighing Solutions in Pipets: The pipet cannot be weighed accurately if it is in contact with any surface of the balance other than the balance pan. For this reason, and to protect against spills and leaks, the pipet is placed tip up in a small paper or plastic cup which sits on the pan of the balance.

Procedure

Part 1: Preparation of the sodium hydroxide solution.

1. Determine the mass of a small beaker (±0.001 g). Add 3-4 sodium hydroxide pellets, quickly close the NaOH container, then reweigh the beaker and pellets. Enter both masses in a Data Table. The mass of NaOH should be on the order of 0.3-0.5 g.

2. Add 5-6 mL of distilled water to the beaker, then swirl the beaker gently to dissolve the pellets. Carefully feel the bottom of the beaker; is the dissolving exothermic or endothermic?

3. Determine the mass of your clean, dry 25-mL graduated cylinder. Record the mass in the Data Table.

4. When the pellets of sodium hydroxide have completely dissolved, pour the solution from the beaker into the graduated cylinder. Use 4-5 mL of distilled water from your wash bottle to rinse the beaker, then transfer the rinsings to the contents of the graduated cylinder. Rinse the beaker again with 4-5 mL of distilled water, and again transfer the rinsings to the graduate. Continue the rinsing and transferring until the total volume of solution is exactly 25.00 mL. Do not go above the 25.00 mL line on the graduate. (Why not?)

5. Determine the total mass of the graduated cylinder and the solution. Be careful to dry the outside and the base of the graduate before you place it on the balance pan.

Part 2: Titration of Vinegar.

1. Rinse and dry your small beaker, then return the standard sodium hydroxide solution to the beaker. Label a clean microtip pipet, "NaOH," then fill it from the solution in the beaker. Weigh the pipet and record the mass in the Data Table.
2. Fill a second microtip pipet, labeled “Vinegar,” or simply, “VIN,” from the commercial vinegar bottle. Weigh the filled pipet and record its mass.

3. Weigh a clean 10-mL Erlenmeyer flask. Transfer some of the vinegar solution to the flask, then reweigh the pipet. The mass of vinegar used should be between 0.8 and 1.2 grams; if it is less than 0.8 g, add a bit more, then reweigh the pipet with the remaining vinegar. Record the mass of the pipet and contents in the Data Table.

4. Add 1 drop of phenolphthalein indicator from the phenolphthalein pipet (labeled “PHTH”) to the contents of the flask and swirl gently.

5. Dropwise and with swirling, add your NaOH solution a few drops at a time until you get a magenta color that does not fade with mixing and that lasts at least 20 seconds. The lighter the pink color, the better. Reweigh the NaOH pipet and record the mass in the Data Table.

6. Repeat the titration three more times, recording the masses of the pipets before and after each trial. You will probably need to refill one or both pipets from time to time. Try to plan so that you don’t have to refill in the middle of a trial.

   **Note:** Use a clean flask for each titration; if necessary, wash flasks between trials, then rinse with distilled water. You need not dry the flasks.

### Cleaning Up

The contents of your titration vessels are safe to be rinsed down the drain with water. However, any unused sodium hydroxide solution remaining in your pipet should be neutralized.

1. Empty the pipet into the beaker or flask containing any unused sodium hydroxide remaining from the experiment and one drop of phenolphthalein.

2. Add vinegar until the pink color of the phenolphthalein disappears; the color change signals that neutralization is complete. Dispose of this neutral solution down the drain with plenty of water.

3. Rinse all equipment thoroughly with water, followed by distilled water, then leave it on paper towels to drain.

4. Wash your hands before leaving the laboratory.

### Analysis and Conclusions

Complete the **Analysis and Conclusions** section for this experiment either on your Report Sheet or in your lab report as directed by your teacher. Show samples of all calculations.

1. Calculate the mass of sodium hydroxide used in preparing your solution. Convert this to moles.
2. Determine the molarity of your sodium hydroxide solution.

3. Determine the mass of the sodium hydroxide solution you prepared, then use that mass to determine the density of your sodium hydroxide solution.

4. Calculate the concentration of the NaOH solution in moles of NaOH per gram of solution.

5. From your data, calculate the mass of NaOH solution used, the number of moles of NaOH used, and the number of moles of acetic acid that must have been present for each of your four titrations. Show your work for trial 1; enter the results for all four titrations in a Data Table.

6. Determine the mass of acetic acid present in each of your four titration samples. Find the percent of acetic acid in each vinegar sample, by dividing the mass of acetic acid present by the mass of vinegar used. Convert the decimal fraction to a percent. Present your results in a Summary Table.

7. Calculate an average value for the mass percent of acetic acid in the vinegar you analyzed. Base your average on the three trials that show the closest agreement; omit the trial that deviates most greatly from the others.

8. Calculate the deviation from the average for each of the four trials, then calculate the average deviation for the three trials that show the best agreement.

9. Report the mass percent of acetic acid in the vinegar as a percent ± average deviation.
   Assuming the density of white vinegar to be 1.0 g/mL, calculate:
   a. the mass of 1.0 L of white vinegar
   b. the mass of acetic acid in 1.0 L of vinegar
   c. the number of moles of acetic acid in 1.0 L of vinegar
   d. the molar concentration (molarity) of acetic acid in white vinegar

**Something Extra**

Sodium hydroxide reacts with the moisture and carbon dioxide in the air, so it is very likely that the mass you reported in question 1 of Analysis and Conclusions is not all sodium hydroxide. Find out how to use potassium acid phthalate, KHP, to standardize a sodium hydroxide solution. Then, with your teacher's permission, repeat the experiment, this time standardizing the solution before proceeding to Part 2.
Analysis of Vinegar

Prelaboratory Questions

1. Write the equation for the reaction between acetic acid and NaOH. Use the structural formula for acetic acid, found in Chapter 16. In the formulas of the reactants, circle the atoms that form water.

2. What is the purpose of the indicator? How does it tell you when a titration is complete?

3. Give two reasons for Safety Special Note #2. (Hint: Consider both your own experiment and that of the person who uses the balance after you.)

4. What does the parenthetical expression (± 0.001 g) mean in Step 1 of Part 1 of the Procedure?

5. Read Step 4 of Part 1 of the Procedure. Answer the question that appears in parentheses.

Data/Observations

Data Table 1

<table>
<thead>
<tr>
<th>Mass of empty beaker</th>
<th>g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of beaker and NaOH pellets</td>
<td>g</td>
</tr>
<tr>
<td>Mass of empty, dry graduated cylinder</td>
<td>g</td>
</tr>
<tr>
<td>Mass of graduated cylinder and NaOH solution</td>
<td>g</td>
</tr>
</tbody>
</table>

Data Table 2

| Mass of Vinegar Pipet (g) | Mass of NaOH Pipet (g) |
Analysis of Vinegar

Before titration 1  ____________ g  Before titration 1  ____________ g
After titration 1  ____________ g  After titration 1  ____________ g

Before titration 2  ____________ g  Before titration 2  ____________ g
After titration 2  ____________ g  After titration 2  ____________ g

Before titration 3  ____________ g  Before titration 3  ____________ g
After titration 3  ____________ g  After titration 3  ____________ g

Before titration 4  ____________ g  Before titration 4  ____________ g
After titration 4  ____________ g  After titration 4  ____________ g

Analysis and Conclusions

Samples of all calculations are to be shown in the spaces provided.

Part 1

1. Calculate the mass of sodium hydroxide used in preparing your solution. Convert this to moles.

2. Determine the molarity of your sodium hydroxide solution.

3. Determine the mass of the sodium hydroxide solution you prepared, then use that mass to determine the density of your sodium hydroxide solution.

4. Calculate the concentration of the NaOH solution in moles of NaOH per gram of solution.

Part 2

Table 3

<table>
<thead>
<tr>
<th>Mass of Vinegar Used (g)</th>
<th>Mass of NaOH Used (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titration 1</td>
<td>____________</td>
</tr>
<tr>
<td>Titration 1</td>
<td>____________</td>
</tr>
</tbody>
</table>
5. From your data, calculate the mass of NaOH solution used, the number of moles of NaOH used, and the number of moles of acetic acid that must have been present for each of your four titrations. Show your work for trial 1; enter the results for all four titrations in Data Table 4.

### Table 4

<table>
<thead>
<tr>
<th>Titration 1</th>
<th>Titration 2</th>
<th>Titration 3</th>
<th>Titration 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaOH solution used (g)</td>
<td>NaOH solution used (g)</td>
<td>NaOH solution used (g)</td>
<td>NaOH solution used (g)</td>
</tr>
<tr>
<td>___________ g</td>
<td>___________ g</td>
<td>___________ g</td>
<td>___________ g</td>
</tr>
<tr>
<td>Moles of NaOH used mol</td>
<td>Moles of NaOH used mol</td>
<td>Moles of NaOH used mol</td>
<td>Moles of NaOH used mol</td>
</tr>
<tr>
<td>___________ mol</td>
<td>___________ mol</td>
<td>___________ mol</td>
<td>___________ mol</td>
</tr>
<tr>
<td>Moles of HC₂H₃O₂ in vinegar sample mol</td>
<td>Moles of HC₂H₃O₂ in vinegar sample mol</td>
<td>Moles of HC₂H₃O₂ in vinegar sample mol</td>
<td>Moles of HC₂H₃O₂ in vinegar sample mol</td>
</tr>
<tr>
<td>___________ mol</td>
<td>___________ mol</td>
<td>___________ mol</td>
<td>___________ mol</td>
</tr>
</tbody>
</table>

6. Determine the mass of acetic acid present in each of your four titration samples. Then find the percent of acetic acid in each vinegar sample, by dividing the mass of acetic acid present by the mass of vinegar used. Convert the decimal fraction to a percent. Present your results in the Summary Table. Show the calculations for Trial 1 in the space below.

### Summary Table

<table>
<thead>
<tr>
<th></th>
<th>Titration 1</th>
<th>Titration 2</th>
<th>Titration 3</th>
<th>Titration 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>mol HC₂H₃O₂</td>
<td>mol</td>
<td>mol</td>
<td>mol</td>
<td>mol</td>
</tr>
</tbody>
</table>
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8. Calculate the deviation from the average for each of the four trials, then calculate the average deviation for the three trials that show the best agreement.

9. Report the mass percent of acetic acid in the vinegar as a percent ± average deviation.

10. Assuming the density of white vinegar to be 1.0 g/mL, calculate:
   a. the mass of 1.0 L of white vinegar;
   b. the mass of acetic acid in 1.0 L of vinegar;
   c. the number of moles of acetic acid in 1.0 L of vinegar; and
   d. the molar concentration (molarity) of acetic acid in white vinegar.

Something Extra

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Find out how to use potassium acid phthalate, KHP, to standardize a sodium hydroxide solution. Then, with your teacher's permission, repeat the experiment, this time standardizing the solution before proceeding to Part 2.