**Experiment:**  
*Preparation and Standardization of a Sodium Hydroxide Solution*

**BACKGROUND**

This is the first of two experiments for which the overall goal is to determine the molar concentration of acetic acid (CH₃CH₂COOH) in vinegar (a solution composed primarily of water and acetic acid). However, before the acetic acid can be analyzed, a solution of sodium hydroxide must be prepared and its molar concentration determined. You will prepare and standardize a solution of NaOH in this experiment. The molar concentration of the NaOH will be determined by the titrating samples of oxalic acid dihydrate of known mass.

\[
\text{Dissolution of oxalic acid dihydrate in water:} \quad \text{H}_2\text{C}_2\text{O}_4\cdot2\text{H}_2\text{O}(s) \rightarrow \text{H}_2\text{C}_2\text{O}_4(aq) + 2 \text{H}_2\text{O}(l)
\]

\[
\text{Titration of the oxalic acid with NaOH:} \quad \text{H}_2\text{C}_2\text{O}_4(aq) + 2 \text{NaOH}(aq) \rightarrow \text{Na}_2\text{C}_2\text{O}_4(aq) + 2 \text{H}_2\text{O}(l)
\]

**PROCEDURE**

**Preparing the NaOH Solution**

1) Using a graduated cylinder, transfer approximately 100 mL of approximately 1 M NaOH solution to a large beaker or flask or bottle (600 mL or larger).
2) With continuous stirring, dilute the solution to a total volume of approximately 500 mL using deionized water.
3) Continue stirring, make certain that the original solution and the added water are completely mixed.
4) Label the solution with its identity and your name(s). This solution will be used in this experiment and the next one. Keep it covered at all times that it is not being used. Be sure to save it for the next experiment, or you will have to repeat this experiment!

**Standardizing the NaOH Solution**

5) Obtain a buret and clean it thoroughly. First rinse thoroughly with water (use soap and water if necessary). Then, add approximately 10 mL of the NaOH solution and use it to thoroughly coat and rinse the buret. Drain the solution fully from the buret.
6) Set up the buret on a ring stand with a double buret clamp (your instructor will demonstrate).
7) Add NaOH solution to the buret so that it is filled up to between the 0 mL and 10 mL marks. Do NOT spend extra time getting it right at 0.00 mL. Record the precise volume reading as the initial volume in your lab notebook. Be sure to read the buret to its full precision.
8) Mass out a sample of oxalic acid dihydrate between 0.23 and 0.27 g and record the precise mass (to the full precision of your balance) in your lab notebook.
9) Transfer the solid to a 125-mL Erlenmeyer flask (or another appropriate piece of glassware). Use a small amount of deionized water to insure that it is all transferred.
10) Add approximately 20 mL of deionized water to the container and stir until the solid is dissolved (or nearly so).
11) Add 2-3 drops of phenolphthalein solution and stir the solution. This indicator will allow you to visualize the endpoint.
12) Begin titrating the sample. Add the sodium hydroxide solution about 1mL at a time for the first 10-12 mL, swirling continuously to keep the solution fully mixed.
13) Begin adding the solution more slowly, still continuously stirring. As soon as you notice any pink color develop when drops hit the solution, begin adding even more slowly – one drop at time, with swirling to dissipate the pink color.
14) When one drop causes the pink color to persist for approximately 30s or longer, you have reached the endpoint. The goal is to achieve lightest pink color possible that persists. Record the final volume from the buret.
15) Repeat steps 7-14 for two additional samples.
16) Using the data acquired and appropriate stoichiometry calculations, determine the [NaOH] for each trial.
17) Calculate the average [NaOH] and the percent relative range for your data, but do not yet record these values in your notebook. Show your results to your instructor. Your instructor will discuss with you whether or not additional trials are necessary.
18) After completing any additional trials required and the corresponding calculations, record the results of your final calculations in your lab notebook.
19) Store your labeled, covered solution for the next experiment.
Formulas:

\[
[X] = \frac{\text{mol } X}{\text{Volume (in L) solution of } X}
\]

Percent Relative Range = \(\frac{\text{High Value} - \text{Low Value}}{\text{Average Value}} \times 100\%\)

Data Table: Standardization of Sodium Hydroxide

<table>
<thead>
<tr>
<th>Trial</th>
<th>1</th>
<th>2</th>
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<th>4</th>
<th>5</th>
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</thead>
<tbody>
<tr>
<td>Mass of oxalic acid dihydrate</td>
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<tr>
<td>Final volume reading of NaOH</td>
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<tr>
<td>Initial volume reading of NaOH</td>
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<td>Volume of NaOH solution added</td>
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<td>Moles of NaOH used (from stoichiometric calculation)</td>
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<td>[NaOH]</td>
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<tr>
<td>Trials used to determine the average [NaOH] and the percent relative range</td>
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<tr>
<td>Average [NaOH]</td>
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<td>Percent Relative Range</td>
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Prepare in your Laboratory Notebook

Prelaboratory Assignment

Prepare a proper HEADING and the following sections:

PURPOSE
Provide a statement of the experimental purpose.

CHEMICAL REACTION
Provide the balanced equation for the reaction carried out in the titration.

DATA TABLES / OBSERVATIONS
Prepare a data table for the experiment using the provided table as your guide.

OTHER INFORMATION as directed by your instructor

Laboratory Report

Prepare a proper HEADING and the following sections:

DATA / OBSERVATIONS / CALCULATIONS / RESULTS
During the experiment, you will collect data in the tables you prepared in your lab notebook. Calculations should be labeled, show the formula used, include units, show work, and the final results should reflect the proper number of significant figures (except in whole number ratios).

CONCLUSIONS
Report the average [NaOH] and its Percent Relative Range.

OTHER INFORMATION as directed by your instructor