Chem 111 – Experiment 2 – Simulation – Standardization of an NaOH Solution

Background

Standardization of an NaOH Solution

Many solutions with a specific concentration are easy to prepare by combining a measured mass of a solid and adding solvent to produce a measured total volume of solution. But in some cases, precisely weighing the solute is not possible. In such cases, the solution is standardized after it is prepared to ensure a specific final molar concentration.

Sometimes when preparing a solution, only the approximate final concentration is known. For example, solid sodium hydroxide (NaOH) is hygroscopic, meaning it absorbs moisture from the atmosphere, and therefore it is difficult to weigh accurately. Depending on the storage conditions, the mass percent of water can vary significantly and there will be less NaOH in a given mass of the solid than expected.

Before an NaOH solution can be used to perform quantitative chemical analyses such as titration, it must be standardized with an acid that is a primary standard. For a substance to be a primary standard, the following criteria should be met:

- It must be available in very pure form.
- It must be reasonably soluble.
- It must be stable in the pure form and in solution.
- It must be non-hygroscopic and easily dried.
- It must be a compound with a reasonably high molar mass to minimize weighing errors.
- It must react rapidly with the substance being standardized with a well-known stoichiometry.

Benzoic acid is a good primary standard because of its high purity, relatively large molar mass, and because it is not hygroscopic. The balanced acid–base reaction with sodium hydroxide is shown below.

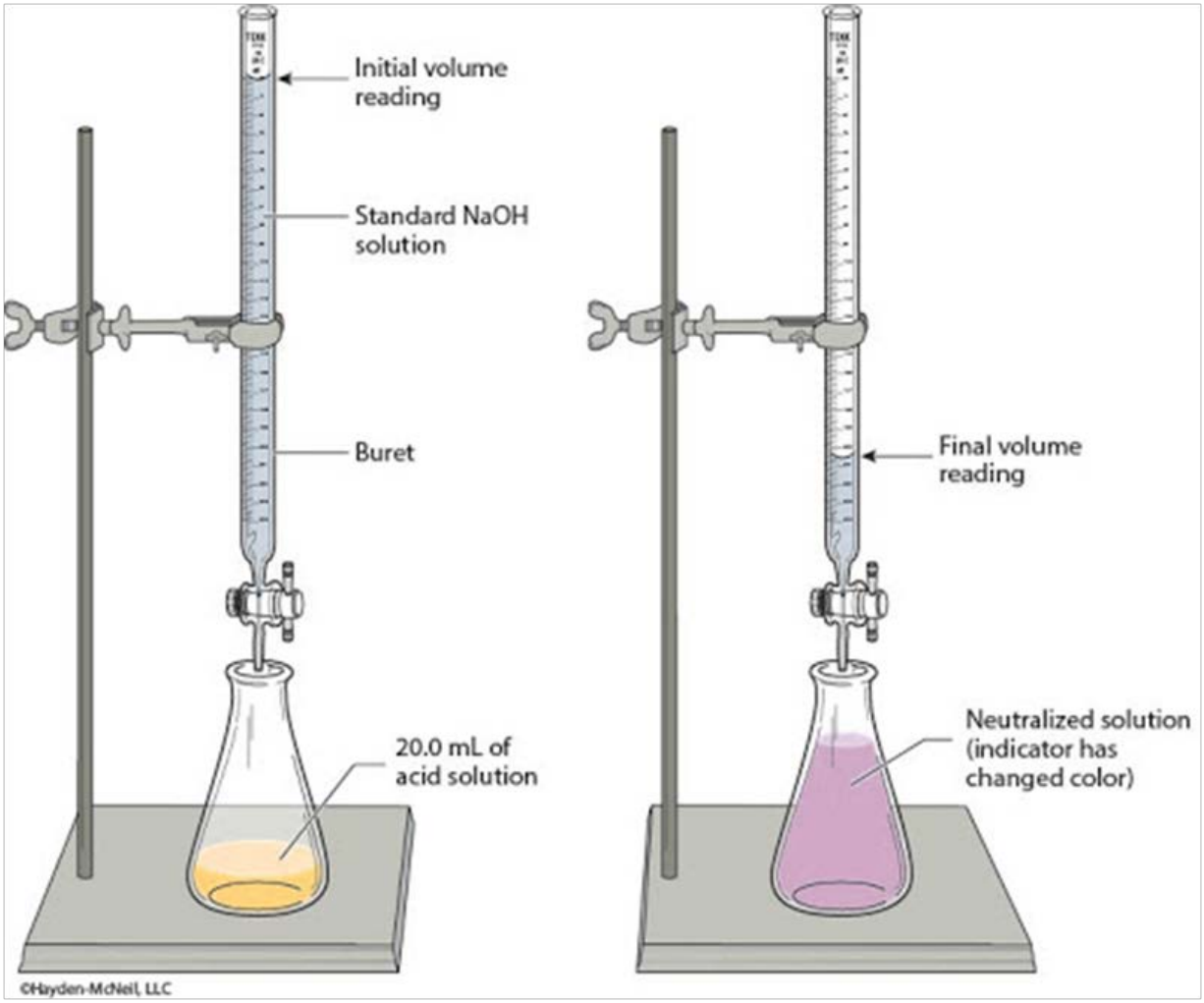
$$HC_7H_5O2(aq) + NaOH(aq) = NaC_7H_5O_2(aq) + H_2O(l)$$

Standardization Titrations

Titration is a volumetric analysis technique used to determine the unknown concentration of a solution by using the known concentration of another. Solutions are often standardized using titration. In an acid–base standardization titration, either a solution of a base of known concentration (titrant) is used to determine the exact concentration of a solution of acid (analyte) or vice versa. However, some standardization titrations use a titrant solution of approximate concentration to titrate a known amount of primary standard.

A color indicator or a pH meter can be used to determine the end point of the titration. The end point of a titration occurs when the indicator changes color, typically when a small excess of the titrant has been added. If the indicator is chosen well, the end point will correspond closely to the point when all the analyte has been neutralized by the titrant (equivalence point). Phenolphthalein is a commonly used acid–base indicator that is

colorless in acidic solutions, but becomes pink when the solution turns basic. A sample titration set up is shown in the figure below.



Acid -Base Titration Set-Up.
An acid sample is titrated with a solution of NaOH using a color-changing indicator.

Molarity Calculations

The number of moles of primary standard used is calculated using the formula below.

$$n_{analyte} = m / MM$$

Where:

n:	is the number of moles of benzoic acid.
m:	is the mass in grams of benzoic acid.
MM:	is its molar mass.
	The molar mass of benzoic acid is 122.12 g/mol.

The number of moles of sodium hydroxide can be calculated from the reaction stoichiometry. For this reaction the sodium hydroxide reacts with benzoic acid in a 1:1 molar ratio.
(Take care here as it is not always 1:1, always check the stoichiometric coefficients in the Balanced Equation.)

 # moles of benzoic acid = # moles of sodium hydroxide

The volume of sodium hydroxide dispensed during the titration is used to determine the molarity of the sodium hydroxide solution.

$$\textbf{Molarity} = \frac{\textbf{moles}}{\textbf{V(L)}}$$

Common mistake is to forget to convert the Volume from mL to L.

Sample Calculation:

An example calculation is shown below for a titration in which **15.32 mL of NaOH** solution was needed to titrate **1.12g benzoic acid** standard.

First convert the 1.12g benzoic acid to moles by dividing by its Molar Mass

$$\textbf{1.12 g benzoic acid} \left| \frac{\textbf{1 mol}}{\textbf{122.12 g}} \right. = \textbf{9.171x10}^{\textbf{-3}}\textbf{ mol benzoic acid}$$

Secondly covert moles of benzoic acid to moles of sodium hydroxide using the balanced chemical equation shown on page 1.

$$\textbf{9.171x10}^{\textbf{-3}}\textbf{ mol benzoic acid} \left| \frac{\textbf{1 mol NaOH}}{\textbf{1 mol benzoic acid}} \right. = \textbf{9.171x10}^{\textbf{-3}}\textbf{ mol NaOH}$$

Finally determine the Molarity of the NaOH using: **M = n / V(L)**

$$\textbf{M} = \frac{\textbf{9.171x10}^{\textbf{-3}}\textbf{ mol NaOH}}{\textbf{0.01532 L}} = \textbf{0.599M}$$

About This Lab

In this lab, you will use dry benzoic acid as the primary acid standard to determine the exact molar concentration of a sodium hydroxide solution. To do this, you will titrate an accurately measured mass of dry benzoic acid with a prepared NaOH solution of approximate concentration. You will use phenolphthalein as an indicator. You will then use the standardized NaOH solution to determine the concentration of a sample of acetic acid using titration.

Open the simulation by clicking on the virtual lab icon shown on the left on the Hayden-McNeil Web Site. The simulation will launch in a new window.

You may need to move or resize the window in order to view both the Procedure and the simulation at the same time.

Follow the instructions in the Procedure to complete each part of the simulation. When instructed to record your observations, record data, or complete calculations, record them for your own records in order to use them later to complete the post-lab assignment.

Procedures

Experiment 2a – Standardize an NaOH Solution Using Benzoic Acid as Primary Standard.

Experiment 2a – Part 1 – Prepare the NaOH Solution

1. Take a **250 mL volumetric flask** from the **Containers shelf** and a **balance** from the **Instruments shelf** and place them on the **workbench**. **Zero** the **mass of the volumetric flask** on the balance.

2. Take **sodium hydroxide** from the **Materials shelf** and add **1.0g** to the flask. **Record the mass** from the balance display.

3. **Place** the **volumetric flask** on the **workbench**. Add **100 mL** of **water** from the **Materials shelf** to the volumetric flask. This dissolves the solid. **Add water again** up to the **maximum volume allowed**. This produces a total volume in the flask of 250.00 mL and the meniscus of the liquid is level with the volume mark on the neck of the flask.
   Note: It is important to dissolve the solid before filling a volumetric flask to the line because the density of the overall solution is different from the densities of the separate solid and liquid. The volumetric flask is designed to contain a specific volume of liquid. In a typical lab, if the liquid level becomes greater than the mark on the flask, the concentration is no longer accurately known and the solution needs to be remade. In the virtual lab there is a limit on the volume of the flask that prevents you from adding liquid above the line.

4. **Calculate the expected concentration** of the sodium hydroxide solution. *The molar mass of NaOH is 39.997 g/mol.*

5. **Calculate and record** the **expected mass of benzoic acid** required to react with **20.00 mL** of a **0.100 M sodium hydroxide solution**. **Record** the **mass of benzoic acid** using **three significant digits** to reference later.

Experiment 2a – Part 2 – Perform a Coarse Titration

1. Take a clean **250 mL Erlenmeyer flask** from the **Containers shelf** and place it on the balance. **Zero the mass of the flask on the balance**.

2. Add the **required mass of benzoic** acid **calculated in step 5 of Part 1** to the Erlenmeyer flask. **Record the mass** value from the balance and **move the flask to the workbench**.

3. Take **water** from the **Materials shelf** and **add 100 mL** to the Erlenmeyer flask. The 100 mL of water should be enough to dissolve the solid benzoic acid.

4. Take **phenolphthalein** from the **Materials shelf** and **add two drops** to the Erlenmeyer flask.

5. Take a **pH meter** from the **Instruments shelf** and **place it into the flask**. **Record the initial pH** of the solution.

6. Take a **burette** from the **Containers shelf** and place it on the **workbench**. Place a **50 mL beaker** from the **Containers shelf** underneath the tip of the burette as a waste container.

7. Take the **prepared sodium hydroxide** solution from the **workbench** and **add 52 mL** to the **burette**. **Click and hold the stopcock** of the buret for about **4 seconds** to drain liquid from the buret into the

waste container until the **titrant level is at or below the 0 mL mark**. **Double-click on the buret to read** and **record the liquid volume at the meniscus**. *This initial volume reading does not need to be exactly 0.00 mL*.

8. Remove the waste container and **move the Erlenmeyer flask onto the base of the burette**.

9. **Perform a coarse titration**, **adding large increments** of titrant (**~2 mL**) by **pressing and holding the stopcock at the bottom of the burette for about 4 seconds**. Pause after each dispensation. Record the volume dispensed.

10. **Check if the end point has passed**. When the reaction reaches the **end point, the solution changes color**. Also, as sodium hydroxide is added to the benzoic acid solution, the pH increases.

11. **Stop once you reach the end point**. **Record** both the **last dispensed volume where the solution was colorless** (right before the end point) and **the first dispensed volume where the solution changed color**. **Record the pH of the solution after the color change**. *You will use the volume dispensed before the end point in the fine titration*.

12. Discard the Erlenmeyer flask by emptying it into the waste then dragging it to the sink.

Experiment 2a – Part 3 – Perform Fine Titrations

1. **Prepare an Erlenmeyer flask as described in steps 1-5 in Part 2 of Experiment 2**. *The use of the pH meter is optional during the fine titrations*.

2. Take the volumetric flask of sodium hydroxide solution and refill the burette to a total volume of about 3 mL less than the maximum volume. Drain the first few milliliters into the waste container until the titrant level is at or below the 0 mL mark. Double-click the burette to read and record the initial volume.

3. Remove the waste container and move the Erlenmeyer flask onto the base of the burette.

4. **Click and hold the stopcock of the burette to quickly add the "before the end point" volume of sodium hydroxide** determined in the coarse titration.

5. Add titrant **one drop at a time** using **single clicks on the stopcock**. This can be tedious, but if you click and hold you might miss the exact end point of the titration. Be sure to pause between each addition.

6. When the **solution changes color**, stop adding titrant. **Record the volume dispensed**.

7. Clear your station by emptying the Erlenmeyer flask into the waste, then placing the flask in the sink.

8. Repeat the **fine titration one more time, and record the results**.

9. **Calculate the concentration of the sodium hydroxide solution**. The rest of the sodium hydroxide solution can now be used in further lab work as a secondary standard with a **reliably known concentration equal to the average of the two titrations**.

Experiment 2b – Use the Standardized NaOH Solution to Determine the Concentration of an Acid.

Experiment 2b – Part 1 – Perform a Coarse Titration

1. Take a **250 mL Erlenmeyer flask** and a **50 mL graduated cylinder** from the **Containers shelf** and place them on the workbench.

2. Take **acetic acid unknown #1** from the **Materials shelf** and **add 25 mL to the graduated cylinder**. **Double-click the graduated cylinder to read and record the exact volume**, then transfer the liquid to the flask.

3. Take **phenolphthalein** from the Materials shelf and add **two drops to the flask**.

4. **Take a pH meter** from the **Instruments shelf** and place it **into the Erlenmeyer flask**. **Record the pH**.

5. **Add about 75 mL** of **water to the Erlenmeyer flask**. This will help make the color change at the end point more visible. **Record the pH of the diluted acetic acid**.

6. Take the NaOH solution from the workbench and refill the burette as described previously. Double-click to read and record the starting volume.

7. Move the Erlenmeyer flask onto the base of the burette.

8. **Perform a coarse titration**, adding large increments of titrant (~2 mL) from the burette by pressing and holding the stopcock at the bottom of the burette. Pause after each dispensation. Record the volume dispensed.

9. Check if the end point has passed. When the reaction reaches the end point, the solution changes color. Also, as more sodium hydroxide is added to the acetic acid solution, the pH increases.

10. Stop once you reach the end point. **Record both the last dispensed volume where the solution was colorless** (right before the end point) and the **first dispensed volume where the solution changed color**. **Record the pH after the color change**. You will use the volume dispensed right before the end point in the fine titration.

11. Clear your station by emptying the Erlenmeyer flask into the waste, then placing the flask in the sink.

Experiment 2b – Part 1 – Perform Fine Titrations.

1. Set up the titration as described in steps 1-7 in Part 1 of Experiment 2. *The pH meter is optional during the fine titration*.

2. **Click and hold the stopcock of the burette** to quickly **add the "before the end point"** volume of sodium hydroxide determined in the coarse titration.

3. Add sodium hydroxide in small increments, down to one drop at a time. This can be tedious, but if you click and hold you might miss the exact end point of the titration.

4. When the **solution changes color**, stop adding titrant. **Record the volume dispensed**.

5. Clear your station by emptying the Erlenmeyer flask into the waste, then placing the flask in the sink.

6. **Repeat the fine titration twice more, and record the results.**
   Note: If you run out of standard solution you will need to repeat Experiment 1. A new standard solution will need to be prepared and analyzed.

7. Clear the bench of all materials, containers, and instruments, then use the **Report File** that you downloaded from the **General Chemistry web site**. When completed **send it to your TA.**