Standardizing a Solution of Sodium Hydroxide

It is often necessary to test a solution of unknown concentration with a solution of a known, precise concentration. The process of determining the unknown's concentration is called *standardization*.

Solutions of sodium hydroxide are virtually impossible to prepare to a precise molar concentration because the substance is hygroscopic. In fact, solid NaOH absorbs so much moisture from the air that a measured sample of the compound is never 100% NaOH. On the other hand, the acid salt potassium hydrogen phthalate, $KHC_8H_4O_4$, can be measured out in precise mass amounts. It reacts with NaOH in a simple 1:1 stoichiometric ratio, thus making it an ideal substance to use to standardize a solution of NaOH.

OBJECTIVES

In this experiment, you will

- Prepare an aqueous solution of sodium hydroxide to a target molar concentration.
- Determine the concentration of your NaOH solution by titrating it with a solution of potassium hydrogen phthalate, abbreviated KHP, with an exact molar concentration.

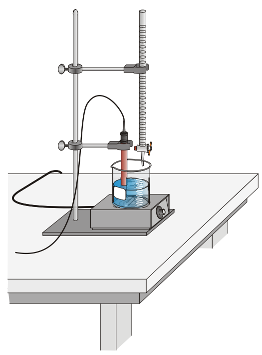

Figure 1

CHOOSING A METHOD

If you choose **Method 1**, you will conduct the titration in a conventional manner. You will deliver volumes of NaOH titrant from a buret. You will enter the buret readings manually to store and graph each pH-volume data pair.

If you choose **Method 2**, you will use a Vernier Drop Counter to conduct the titration. NaOH titrant is delivered drop by drop from the reagent reservoir through the Drop Counter slot. After the drop reacts with the reagent in the beaker, the volume of the drop is calculated and a pH-volume data pair is stored.

MATERIALS

Materials for *both* Method 1 (buret) *and* Method 2 (Drop Counter)

LabQuest	magnetic stirrer
LabQuest App	stirring bar or Microstirrer
Vernier pH Sensor	wash bottle
weighing dish or weighing paper	distilled water
solid potassium hydrogen phthalate	ring stand
solid sodium hydroxide	utility clamp
pipet bulb or pump	250 mL beaker
250 mL Erlenmeyer flask	100 mL graduated cylinder
balance (± 0.01 g)	

Materials required *only* for Method 1 (buret)

50 mL buret	buret clamp

Materials required *only* for Method 2 (Drop Counter)

Vernier Drop Counter	100 mL beaker
60 mL reagent reservoir	10 mL graduated cylinder
a second 250 mL beaker	

PRE-LAB EXERCISE

1. Calculate the mass of sodium hydroxide needed to prepare 100 mL of a 0.10 M solution.

2. Calculate the mass of KHP needed to react completely with 25 mL of a 0.10 M NaOH solution. Consider the reaction equation to be as shown below.

$$HP^{-}(aq) + OH^{-}(aq) \rightarrow H_2O(l) + P^{2-}(aq)$$

METHOD 1: Measuring Volume Using a Buret

1. Obtain and wear goggles.

2. Measure out 100 mL of distilled water into a 250 mL Erlenmeyer flask.

3. Measure out the mass of NaOH that is needed to prepare 100 mL of a 0.10 M solution and add it to the flask of distilled water. Swirl the flask to dissolve the solid. **CAUTION:** *Sodium hydroxide solution is caustic. Avoid spilling it on your skin or clothing*.

4. Measure out the mass of KHP that will completely neutralize 25 mL of 0.10 M NaOH solution. Dissolve the KHP in about 50 mL of distilled water in a 250 mL beaker. Place the beaker of KHP solution on a magnetic stirrer and add a stirring bar. If no magnetic stirrer is available, stir the reaction mixture with a stirring rod during the titration.

5. Connect the pH Sensor to LabQuest and choose New from the File menu. If you have an older sensor that does not auto-ID, manually set up the sensor.

6. Set up a ring stand, buret clamp, and buret to conduct a titration (see Figure 1). Rinse and fill the buret with the NaOH solution.

7. Use a utility clamp to suspend the pH Sensor on a ring stand as shown in Figure 1. Position the pH Sensor in the KHP solution and adjust its position so that it is not struck by the stirring bar. Gently stir the beaker of solution.

8. On the Meter screen, tap Mode. Change the data-collection mode to Events with Entry. Enter the Name (Volume) and Unit (mL).
9. Collect titration data. Conduct the titration carefully, as described below.
   a. Start data collection.
   b. Before you have added any of the NaOH solution, tap Keep and enter **0** as the buret volume in mL. Select OK to store the first data pair.
   c. Add the next increment of NaOH titrant (enough to raise the pH about 0.15 units). When the pH stabilizes, tap Keep, and enter the current buret reading as precisely as possible. Select OK to store the second data pair.
   d. When a pH value of approximately 6.0 is reached, change to 1–3 drop increments. Enter a new buret reading after each increment. At about pH 6.7, add NaOH one drop at a time.
   e. After a pH value of approximately 10 is reached, again add larger increments that raise the pH by about 0.15 pH units, and enter the buret reading after each increment.
   f. Continue adding NaOH solution until the pH value remains constant.
10. Stop data collection to view a graph of pH *vs.* volume. Dispose of the reaction mixture as directed. Rinse the pH Sensor with distilled water in preparation for a second titration.
11. Examine your titration data to identify the region where the pH made the greatest increase. The equivalence point is in this region.
    a. To examine the data pairs on the displayed graph, select any data point.
    b. As you move the examine line, the pH and volume values of each data point are displayed to the right of the graph.
    c. Identify the equivalence point as precisely as possible and record this information.
    d. Store the data from the first run by tapping the File Cabinet icon.
12. An alternate way of determining the precise equivalence point of the titration is to take the first and second derivatives of the pH-volume data.

    Determine the peak value on the first derivative *vs.* volume plot.

    a. Tap the Table tab and choose New Calculated Column from the Table menu.
    b. Enter d1 as the Calculated Column Name. Select the equation 1st Derivative (Y,X). Use Volume as the Column for X and pH as the Column for Y. Select OK.
    c. On the displayed plot of d1 *vs.* volume, examine the graph to determine the volume at the peak value of the first derivative.

    Determine the zero value on the second derivative *vs.* volume plot.

    d. Tap Table and choose New Calculated Column from the Table menu.
    e. Enter d2 as the Calculated Column Name. Select the equation 2nd Derivative (Y,X). Use Volume as the Column for X and pH as the Column for Y. Select OK.
    f. On the displayed plot of d2 *vs.* volume, examine the graph to determine the volume when the 2nd derivative equals approximately zero.
13. Repeat the titration with a second KHP solution. Analyze the titration results in a manner similar to your first trial and record the equivalence point.
14. Print the graph directly from LabQuest, if possible. Alternately, transfer the data to a computer, using Logger *Pro* software. Print a copy of the graph of each titration.

DATA TABLE

Trial	Equivalence point (mL)
1	
2	
3	

DATA ANALYSIS

1. Calculate the molar amount of KHP used to neutralize the NaOH solution.

2. Calculate the molar concentration of the NaOH solution that you prepared.

3. Compare the actual molarity of your NaOH solution with your goal of 0.10 M.