

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, $\text{KHC}_8\text{H}_4\text{O}_4$, 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.

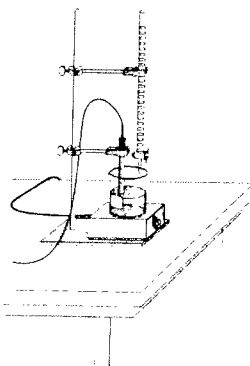


Figure 1

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)

Vernier computer interface	magnetic stirrer
computer	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)

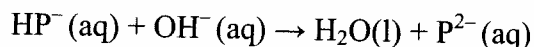
50 mL buret	buret clamp
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Materials required only for Method 2 (Drop Counter)

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.



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 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. Connect a pH Sensor to Channel 1 of a Vernier computer interface. Connect the interface to the computer with the proper cable.
5. Start the *Logger Pro* program on your computer. Open the file "06a NaOH" from the *Advanced Chemistry with Vernier* folder.
6. 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.
7. 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.

8. 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.
9. 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.
10. You are now ready to begin the titration.
 - a. Before adding NaOH titrant, click . Once the displayed pH reading has stabilized, click . In the edit box, type 0 (for 0 mL added). Press the ENTER key to store the first data pair for this experiment.
 - b. Add an increment of NaOH titrant (enough to raise the pH about 0.15 units). When the pH stabilizes, again click . In the edit box, type the current buret reading, to the nearest 0.01 mL. Press ENTER. You have now saved the second data pair for the experiment.
 - c. Continue adding NaOH solution in increments that raise the pH by about 0.15 units and enter the buret reading after each increment.
 - 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 level after each increment.
 - f. Continue adding NaOH solution until the pH value remains constant.
11. When you have finished collecting data, click . Dispose of the reaction mixture as directed. Rinse the pH Sensor with distilled water in preparation for a second titration.
12. Follow the steps below to find the *equivalence point*, which is the largest increase in pH upon the addition of a very small amount of NaOH solution. A good method of determining the precise equivalence point of the titration is to take the second derivative of the pH-volume data, a plot of $\Delta^2\text{pH}/\Delta\text{vol}^2$.
 - a. View a plot of the second derivative on Page 3 by clicking on the Next Page button, .
 - b. Analyze the second derivative plot and record the volume of NaOH at the equivalence point.
13. Return to the original titration graph. Print a copy of the graph and the data set. If you wish to save the results of the first titration, choose Store Latest Run from the Experiment menu.
14. Repeat the titration with a second KHP solution. Analyze the titration results in a manner similar to your first trial and record the volume of NaOH at the equivalence point.

METHOD 2: Measuring Volume with a Drop Counter

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 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. Connect the pH Sensor to CH 1 of the Vernier computer interface. Lower the Drop Counter onto a ring stand and connect its cable to DIG/SONIC 1 of the interface (see Figure 2).

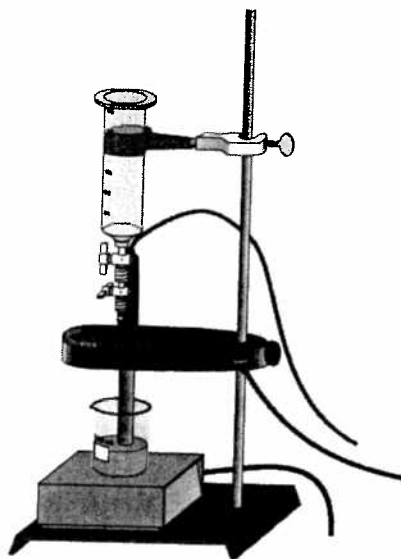


Figure 2

5. Start the *Logger Pro* program on your computer. Open the file "06b NaOH(Drop)" from the *Advanced Chemistry with Computers* folder.
6. Measure out the mass of KHP that will completely neutralize 10 mL of 0.10 M NaOH solution. Dissolve the KHP in about 25 mL of distilled water in a 100 mL beaker.
7. Obtain the plastic 60 mL reagent reservoir. Close both valves by turning the handles to a horizontal position. Follow the steps below to set up the reagent reservoir for the titration.
 - a. Rinse the reagent reservoir with a few mL of the 0.10 M NaOH solution and pour the NaOH into an empty 250 mL beaker.
 - b. Use a utility clamp to attach the reservoir to the ring stand.
 - c. Fill the reagent reservoir with slightly more than 60 mL of the 0.10 M NaOH solution.
 - d. Place the 250 mL beaker, which contains the rinse NaOH, beneath the tip of the reservoir.
 - e. Drain a small amount of NaOH solution into the 250 mL beaker so it fills the reservoir's tip. To do this, turn both valve handles to the vertical position for a moment, then turn them both back to horizontal.
 - f. Discard the drained NaOH solution in the 250 mL beaker as directed.
8. Calibrate the drops that will be delivered from the reagent reservoir. **Note:** If you are using the stored calibration (28 drops per mL), then skip this step.
 - a. Choose Calibrate ► DIG 1: Drop Counter from the Experiment menu.
 - b. Proceed by one of these methods:
 - If you have previously calibrated the drop size of your reagent reservoir and want to continue with the same drop size, select the Manual button, enter the number of Drops/mL, and click . Then proceed directly to Step 9.
 - If you want to perform a new calibration, select the Automatic button, and continue with this step.
 - c. Place a 10 mL graduated cylinder directly below the slot on the Drop Counter, lining it up with the tip of the reagent reservoir.
 - d. Open the bottom valve on the reagent reservoir (vertical). Keep the top valve closed.

- e. Click the Start button on the dialog box.
 - f. Slowly open the top valve of the reagent reservoir so that drops are released at a slow rate (~1 drop every two seconds). You should see the drops being counted on the computer screen.
 - g. When the volume of NaOH solution in the graduated cylinder is between 9 and 10 mL, close the bottom valve of the reagent reservoir.
 - h. Enter the precise volume of NaOH in the edit box. Record the number of Drops/mL displayed on the screen for possible future use.
 - i. Click . Discard the NaOH solution in the graduated cylinder as directed, and set the graduated cylinder aside.
9. Assemble the apparatus.
- a. Place the magnetic stirrer on the base of the ring stand.
 - b. Insert the pH Sensor through the large hole in the Drop Counter.
 - c. Attach the Microstirrer to the bottom of the pH Sensor. Rotate the paddle wheel of the Microstirrer, and make sure that it does not touch the bulb of the pH Sensor.
 - d. Adjust the positions of the Drop Counter and reagent reservoir so they are both lined up with the center of the magnetic stirrer.
 - e. Lift up the pH Sensor, and slide the 100 mL beaker containing the KHP solution onto the magnetic stirrer. Lower the pH Sensor into the beaker.
 - f. Adjust the position of the Drop Counter so that the Microstirrer on the pH Sensor is just touching the bottom of the beaker.
 - g. Adjust the reagent reservoir so its tip is just above the Drop Counter slot.
10. Turn on the magnetic stirrer so that the Microstirrer is stirring at a fast rate.
11. You are now ready to begin collecting data. Click . No data will be collected until the first drop goes through the Drop Counter slot. Fully open the bottom valve. The top valve should still be adjusted so drops are released at a rate of about 1 drop every 2 seconds. When the first drop passes through the Drop Counter slot, check the data table to see that the first data pair was recorded.
12. Continue watching your graph to see when a large increase in pH takes place—this will be the equivalence point of the reaction. When this jump in pH occurs, let the titration proceed for about 3 more milliliters of NaOH solution, then click . Turn the bottom valve of the reagent reservoir to a closed (horizontal) position.
13. Dispose of the reaction mixture as directed.
14. Follow the steps below to find the *equivalence point*, which is the largest increase in pH upon the addition of a very small amount of NaOH solution. A good method of determining the precise equivalence point of the titration is to take the second derivative of the pH-volume data, a plot of $\Delta^2\text{pH}/\Delta\text{vol}^2$.
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15. Return to the original titration graph. Print a copy of the graph and the data set. If you wish to save the results of the first titration, click on Store Latest Run in the Experiment menu.
16. 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.

DATA TABLE

Trial	Equivalence point (mL)
1	
2	

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.