# Standardization of a Primary Standard & Determination of Concentration by Acid-Base Titration

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 once it has been dried in an oven. 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. At the equivalence point, moles acid = moles base = moles salt formed. Since the ratio of reactants is 1:1, the neutralization equation, $M_AV_A = M_BV_B$, also applies since it is just another statement of *moles acid = moles base = moles salt formed* at the equivalence point.

## OBJECTIVES

In this experiment, you will

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

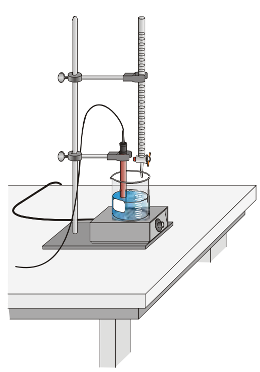

*Figure 1*

## MATERIALS

| | |
|---|---|
| Data Collection Mechanism | solid sodium hydroxide, NaOH |
| pH Sensor | solid potassium hydrogen phthalate, KHP |
| ring stand | distilled water |
| 250 mL beaker | plastic weighing dish or weighing paper |
| 250 mL Erlenmeyer flask | balance (± 0.01 g) |
| 50 mL and 100 mL graduated cylinders | utility clamp |
| magnetic stirrer and stirring bar | buret and buret clamp |
| | 100 mL graduated cylinder |

## Part I: STANDARDIZATION OF PRIMARY STANDARD PROCEDURE

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

2. Set up the data collection system.
   a. Connect the pH Sensor to the interface.
   b. Start the data collection program.
   c. Set up data collection for Events with Entry mode.

3. Measure out the mass of NaOH that is needed to prepare a 0.100 *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 neutralize 25 mL of 0.100 *M* NaOH solution. Dissolve the *analyte,* the KHP, in about 50 mL of distilled water in a 250 mL beaker.

5. Set up a ring stand, buret clamp, and buret to conduct a titration (see *Figure 1*). Place a utility clamp on the ring stand to hold the pH Sensor in place during the titration.

6. Rinse and fill the buret with the *titrant*, the NaOH solution. Place a magnetic stirring bar in the beaker (if available) and place the beaker of KHP solution on a magnetic stirrer under the buret. Connect the pH Sensor to the utility clamp so that the tip of the sensor in immersed in the KHP solution but does not interfere with the movement of the magnetic stirring bar.

7. Conduct the titration carefully. When you have completed the titration, dispose of the reaction mixture as directed. Before conducting a second titration record the results of your first titration, either save the electronic data, or record your data in a table or chart.

8. Repeat the titration with a second KHP solution. Analyze the titration results and record the equivalence point. Save the results of the second titration.

9. Use your titration data to determine 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 $\Delta^2$pH/$\Delta$vol$^2$.

You may use another method to analyze titration data, called a *Gran Plot*. Proposed in the early 1950s by G. Gran, this method uses the reciprocal of ΔpH of the titration data (where ΔpH = pH value – previous pH value). The graph of volume of 1/ΔpH *vs*. titrant volume resembles a V-shaped plot. The inflection point of this plot is the equivalence point volume of the titration.

To use this method, you will first need to create a new calculated column, 1/ΔpH. You can do this in your calculator or with computer graphing software such as Logger *Pro.*

On your resulting plot of 1/ΔpH *vs*. volume (see *Figure 2*), interpolate to find the intersection of two best-fit regression lines. The precise volume where the two linear fits intersect will be the equivalence point volume. In the sample graph shown here, the equivalence point is 10.58 mL.

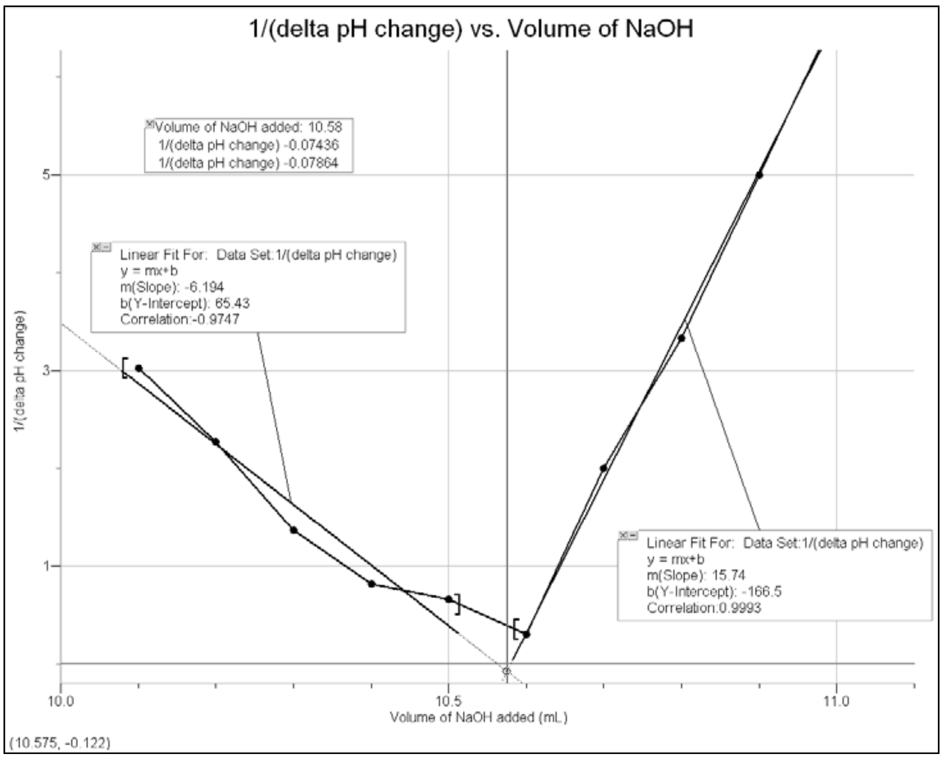


*Figure 2*

## DATA TABLE

| Trial | Equivalence point (mL) |
|---|---|
| 1 | |
| 2 | |

## PART I: PRE-LAB QUESTIONS

1. Calculate the mass of sodium hydroxide needed to prepare 100 mL of a 0.100 *M* solution.

2. Calculate the mass of KHP needed to react completely with 25 mL of a 0.100 *M* NaOH solution. Consider the reaction equation to be as shown below.

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

3. Calculate the molarity of a solution of sodium hydroxide if 23.64 mL of this solution is needed to neutralize 0.5632 g of KHP.

4. It is found that 24.68 mL of 0.1165 *M* NaOH is needed to titrate 0.2931 g of an unknown monoprotic acid to the equivalence point. Calculate the molar mass of the acid.

5. The following data was collected for the titration of 0.145 g of a weak monoprotic acid with 0.100 *M* NaOH as the titrant:

| **Volume of NaOH added, mL** | 0.00 | 5.00 | 10.00 | 12.50 | 15.00 | 20.00 | 24.00 | 24.90 | 25.00 | 26.00 | 30.00 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **pH** | 2.88 | 4.15 | 4.58 | 4.76 | 4.93 | 5.36 | 6.14 | 7.15 | 8.73 | 11.29 | 11.96 |

(a) Use computer graphing software to graph pH vs. volume of NaOH.

(b) Analyze your graph. What is volume of NaOH required to reach the equivalence point?

(c) What is the pH at the equivalence point?

(d) Calculate the molar mass of the weak monoprotic acid?

(e) Give the $K_a$ and p$K_a$ value of the acid. Justify your answer.

## PART I: POST-LAB QUESTIONS AND DATA ANALYSIS

1. Graph your data.

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

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

4. Compare the actual molarity of your NaOH solution with your goal of 0.100 *M*.

5. A student fails to wash the weighing paper when transferring the KHP sample into the beaker. What effect does this error have on the calculated molarity of the NaOH solution? Mathematically justify your answer.

6. A student failed to notice an air bubble trapped in the tip of the buret during their experiment. What effect does this error have on the calculated molarity of the NaOH solution? Mathematically justify your answer.

# Acid-Base Titration

A titration is a process used to determine the volume of a solution that is needed to react with a given amount of another substance. In this experiment, your goal is to determine the molar concentration of two acid solutions by conducting titrations with the standardized NaOH solution you mixed in the previous lab exercise. You will be testing a strong acid, HCl, solution and a weak acid, $HC_2H_3O_2$, solution. The reaction equations are shown below in net ionic form.

$$H^+ (aq) + OH^- (aq) \rightarrow H_2O(l)$$

$$HC_2H_3O_2(aq) + OH^- (aq) \rightarrow H_2O(l) + C_2H_3O_2^- (aq)$$

The stoichiometry of the two reactions is identical; thus, your calculations will be straightforward. However, you will observe a significant difference in how the two acid solutions react with NaOH.

In this experiment, you will monitor pH as you titrate. The region of most rapid pH change will then be used to determine the equivalence point. The volume of NaOH titrant used at the equivalence point will be used to determine the molarity of the HCl solution.

## OBJECTIVES

In this experiment, you will

- Accurately conduct acid-base titrations.
- Determine the equivalence point of a strong acid-strong base titration.
- Determine the equivalence point of a weak acid-strong base titration.
- Calculate the molar concentrations of two acid solutions.

## MATERIALS

Data Collection Mechanism
pH Sensor
ring stand
250 mL beaker
magnetic stirrer and stirring bar
25 mL graduated cylinder
50 mL graduated cylinder

YOUR $\simeq$ 0.100 *M* standardized NaOH solution
hydrochloric acid, HCl, solution, unknown concentration
acetic acid, $HC_2H_3O_2$, solution, unknown concentration
distilled water
50 mL buret and buret clamp
utility clamp

## PART II: ACID-BASE TITRATION PROCEDURE

1. Obtain and wear goggles.

2. Add 50 mL of distilled water to a 250 mL beaker. Obtain about 10 mL of a hydrochloric acid solution of unknown concentration from the dispensing buret your instructor has set up. *Record the exact volume of* HCl *that you obtained.* **CAUTION:** *Handle the hydrochloric acid with care. It can cause painful burns if it comes in contact with the skin.*

3. Place the beaker 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.

4. Set up the data collection system.
   a. Connect a pH Sensor to the interface.
   b. Start the data collection program.
   c. Set up data collection for Events with Entry mode.

5. Rinse and fill the buret with your *titrant*, the standardized NaOH solution of known concentration (about 0.100 *M*). **CAUTION:** *Sodium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.*

6. Use a utility clamp to suspend the pH Sensor on the ring stand (see Figure 1). Position the pH Sensor so that its tip is immersed in the HCl solution but is not struck by the stirring bar. Gently stir the beaker of acid solution.

7. Conduct the titration carefully. Listed below is a suggested method of running a titration.
   a. Record an initial pH reading, before adding any $\simeq$ 0.100 *M* NaOH solution.
   b. Add NaOH in small increments that raise the pH of the mixture by about 0.15 units at a time, until you reach a pH of about 3.5.
   c. At this point, add NaOH drop by drop. Continue in this manner until you have recorded data through the equivalence point.
   d. When the pH of the mixture is about 10, add larger increments of NaOH that raise the pH by about 0.15 units, or 1 mL of NaOH at a time, until the pH remains constant.

8. When you have finished the titration, dispose of the reaction mixture as directed. Rinse the pH Sensor with distilled water in preparation for a second titration. Before conducting the second titration, **record your data electronically, or in a table or chart**.

9. Use your titration data to determine the *equivalence point*, which is the largest increase in pH upon the addition of a very small amount of NaOH solution.

10. Print and save the graph and data set of pH *vs*. volume for the HCl trials.

11. Repeat the necessary steps to test the acetic acid solution and analyze the titration data. Print and save the graph and data set for the acetic acid titrations.

## PART II: DATA TABLE

| HCl Trial | Volume HCl (mL) | Equivalence point (mL) |
|---|---|---|
| 1 | | |
| 2 | | |

| $CH_3COOH$ Trial | Volume $CH_3COOH$ (mL) | Equivalence point (mL) |
|---|---|---|
| 1 | | |
| 2 | | |

## PART II: POST LAB QUESTIONS AND DATA ANALYSIS

1. Calculate the molar amounts of NaOH used in the reaction with the HCl solution and with the $HC_2H_3O_2$ solution.

2. Calculate the molar concentration (molarity) of the HCl solution and the $HC_2H_3O_2$ solution.

3. Compare the actual molar concentrations of your two acid solutions with your calculated molarities. Were the calculated molarities of your acid solutions within a reasonable range (about 5%) of the actual values? If not, suggest reasons for the inaccuracy.

4. The equivalence points of the two titration curves were not in the same pH range. Explain.