Acid-Base Titration

A titration is a process used to determine the volume of a solution needed to react with a given amount of another substance. In this experiment, you will titrate hydrochloric acid solution, HCl, with a basic sodium hydroxide solution, NaOH. The concentration of the NaOH solution is given and you will determine the unknown concentration of the HCl. Hydrogen ions from the HCl react with hydroxide ions from the NaOH in a one-to-one ratio to produce water in the overall reaction:

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

When an HCl solution is titrated with an NaOH solution, the pH of the acidic solution is initially low. As base is added, the change in pH is quite gradual until close to the equivalence point, when equimolar amounts of acid and base have been mixed. Near the equivalence point, the pH increases very rapidly, as shown in Figure 1. The change in pH then becomes more gradual again, before leveling off with the addition of excess base.

In this experiment, you will use a pH Sensor to 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.

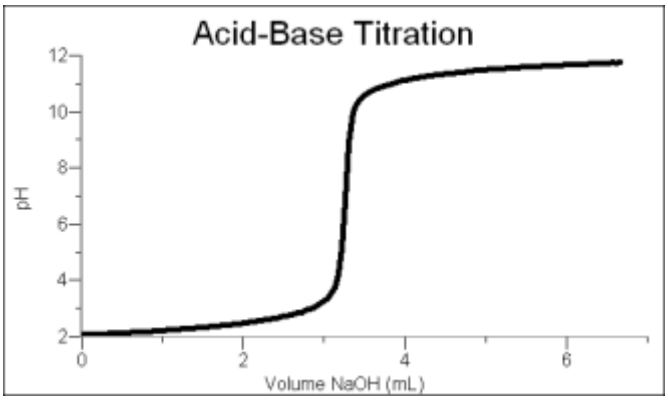

Figure 1

OBJECTIVES

- Use a pH Sensor to monitor changes in pH as sodium hydroxide solution is added to a hydrochloric acid solution.
- Plot a graph of pH *vs*. volume of sodium hydroxide solution added.
- Use the graph to determine the equivalence point of the titration.
- Use the results to calculate the concentration of the hydrochloric acid solution.

MATERIALS

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

Chromebook, computer, **or** mobile device
Graphical Analysis app
Vernier data-collection interface
pH Sensor
Stir Station
magnetic stirring bar
(optional) Phenolphthalein
HCl solution, unknown concentration
~0.1 M NaOH solution
pipet bulb or pump
250 mL beaker
wash bottle
distilled water

Materials required only for Method 1 (buret)

Electrode Support
50 mL buret
10 mL pipet
buret clamp or utility clamp
2nd 250 mL beaker

Materials required only for Method 2 (Drop Counter)

Vernier Drop Counter
60 mL reagent reservoir
5 mL pipet or graduated 10 mL pipet
100 mL beaker
10 mL graduated cylinder
utility clamp

CHOOSE A METHOD

Method 1: Deliver volumes of NaOH titrant from a buret. After titrant is added, and pH values have stabilized, the student is prompted to enter the buret reading manually and a pH-volume data pair is stored.

Method 2: Use a Vernier Drop Counter to take volume readings. 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.

METHOD 1: Measuring Volume Using a Buret

1. Obtain and wear goggles.

2. Use a pipet bulb (or pipet pump) to pipet 10 mL of the HCl solution into a 250 mL beaker. Add 50 mL of distilled water. **DANGER**: *Hydrochloric acid solution,* HCl: *Causes severe skin and eye damage. Do not breathe mist, vapors, or spray. May cause respiratory irritation. May be harmful if swallowed*.

3. Place the beaker on a Stir Station and add a stirring bar.

4. Connect the pH Sensor to the data-collection interface, and then connect the interface to your Chromebook, computer, or mobile device. Launch Graphical Analysis.

5. Set up the data-collection mode.

   a. Click or tap Mode to open Data Collection Settings. Change Mode to Event Based.

   b. Enter **Volume** as the Event Name and **mL** as the Units. Click or tap Done.

6. Use an Electrode Support to suspend a pH Sensor on a Stir Station (see Figure 2). Position the pH Sensor in the HCl solution and adjust its position so it will not be struck by the stirring bar. Turn on the Stir Station, and adjust it to a medium stirring rate (with no splashing of solution). Check to see that the pH value is between 1.5 and 2.5.

Figure 2

7. Obtain a 50 mL buret and rinse the buret with a few mL of the ~0.1 M NaOH solution. Dispose of the rinse solution as directed by your teacher. **WARNING**: *Sodium hydroxide solution,* NaOH: *Causes skin and eye irritation.*

   Use a buret clamp or a utility clamp to attach the buret to the Stir Station as shown in Figure 2. Fill the buret a little above the 0.00 mL level of the buret with ~0.1 M NaOH solution. Drain a small amount of NaOH solution so it fills the buret tip *and* leaves the NaOH at the 0.00 mL level of the buret. Record the precise concentration of the NaOH solution in your data table.

8. You are now ready to perform the titration. This process is faster if one person manipulates and reads the buret while another person enters volumes.

   a. Click or tap Collect to start data collection.

   b. Before you have added any drops of NaOH solution, click or tap Keep and enter **0** as the buret volume in mL. Click or tap Keep Point to store the first data pair for this experiment.

   c. Add the next increment of NaOH titrant (enough to raise the pH about 0.15 units). When the pH stabilizes, click or tap Keep, enter the current buret reading (to the nearest 0.01 mL), and then click or tap Keep Point.

   d. Continue adding NaOH solution in increments that raise the pH by about 0.15 units and enter the buret reading after each increment. When a pH value of approximately 3.5 is reached, change to a one-drop increment. Enter a new buret reading after each increment. **Note**: It is important that all increment volumes in this part of the titration be equal; that is, one-drop increments.

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.

9. Click or tap Stop to stop data collection.

10. Examine the data on the graph of pH *vs*. volume to find the *equivalence point*—that is the largest increase in pH upon the addition of 1 drop of NaOH solution. Move to the region of the graph with the largest increase in pH (you can adjust the Examine line by dragging the flag). Find the NaOH volume just *before* this jump. Record this value in the data table. Then record the NaOH volume *after* the drop producing the largest pH increase was added. **Note**: Another method for determining the equivalence-point volume is described in the Alternate Equivalence Point Method of this experiment.

11. (optional) Export, download, or print a copy of the graph of pH *vs*. volume.

12. Dispose of the beaker contents as directed by your teacher. Rinse the pH Sensor and return it to the pH storage solution.

METHOD 2: Measuring Volume with a Drop Counter

1. Obtain and wear goggles.

2. Add 40 mL of distilled water to a 100 mL beaker. Use a pipet bulb (or pipet pump) to pipet 5.00 mL of the HCl solution into the 100 mL beaker with distilled water. **DANGER**: *Hydrochloric acid solution,* HCl: *Causes severe skin and eye damage. Do not breathe mist, vapors, or spray. May cause respiratory irritation. May be harmful if swallowed*.

3. Obtain approximately 40 mL of ~0.1 M NaOH solution in a 250 mL beaker. Record the precise NaOH concentration in your data table. **WARNING**: *Sodium hydroxide solution,* NaOH: *Causes skin and eye irritation.*

4. Obtain the plastic 60 mL reagent reservoir. **Note**: The bottom valve will be used to open or close the reservoir, while the top valve will be used to finely adjust the flow rate. For now, close both valves by turning the handles to a horizontal position.

   Rinse the reagent reservoir with a few mL of the ~0.1 M NaOH solution. Attach the reagent reservoir to the Stir Station. Add the remainder of the NaOH solution to the reagent reservoir.

   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.

5. Connect the pH Sensor and the Drop Counter to the data-collection interface and then connect the interface to your Chromebook, computer, or mobile device. Launch Graphical Analysis.

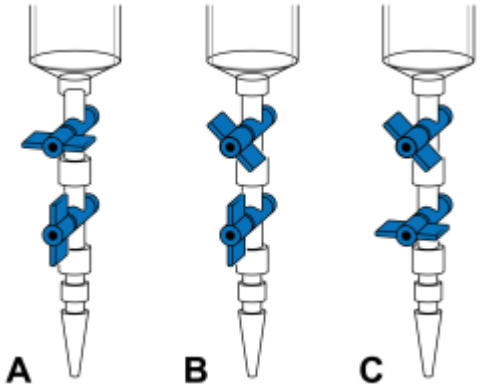

Figure 3

Figure 4

6. Calibrate the Drop Counter so that a precise volume of titrant is recorded in units of milliliters.
   a. Attach the Drop Counter to the Stir Station.
   b. Adjust the handles on the reagent reservoir so the top valve is closed (horizontal) and the bottom valve is open (vertical) (see Figure 3A).
   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. Click or tap the Volume meter and choose Calibrate.
   e. Follow the on-screen prompts to calibrate the Drop Counter. To adjust the drop flow, slowly open the top valve of the reagent reservoir (see Figure 3B) so that drops are released at a slow rate (~1 drop every two seconds). When the volume of solution in the graduated cylinder is between 9 and 10 mL close the bottom valve (see Figure 3C).
   f. Discard the solution in the graduated cylinder as indicated by your instructor and set the graduated cylinder aside.
7. Assemble the apparatus.
   a. Insert the pH Sensor through the large hole in the Drop Counter.
   b. Adjust the positions of the Drop Counter and reagent reservoir so they are both lined up with the center of the Stir Station.
   c. Lift up the pH Sensor, and slide the beaker containing the HCl solution onto the Stir Station. Lower the pH Sensor into the beaker. Check to see that the pH value is between 1.5 and 2.5.
   d. Place the stirring bar in the beaker and adjust the position of the pH Sensor so that it will not be struck by the stirring bar.
   e. Adjust the reagent reservoir so its tip is just above the Drop Counter slot.
8. Turn on and adjust the Stir Station so it is stirring at a fast rate.

9. You are now ready to begin collecting data. Click or tap Collect to start data collection. 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 graph to see that the first data pair was recorded.

10. 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 several more milliliters of titrant, then click or tap Stop to stop data collection. Turn the bottom valve of the reagent reservoir to a closed (horizontal) position.

11. Dispose of the beaker contents as directed by your teacher.

12. Examine the data on the graph of pH *vs*. volume to find the *equivalence point*. Move to the region of the graph with the largest increase in pH. Find the NaOH volume just *before* this jump. Record this value in the data table. Then record the NaOH volume *after* the drop producing the largest pH increase was added. **Note**: Another method for determining the equivalence-point volume is described in the Alternate Equivalence Point Method of this experiment.

13. (optional) Export, download, or print the graph.

14. If time permits, repeat the procedure.

ALTERNATE EQUIVALENCE POINT METHOD

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.

1. Determine the peak value on the first derivative *vs*. volume plot.
   a. Click or tap Column Options, ⋯, in the pH column header in the table. Then, choose Add Calculated Column.
   b. Enter **d1** as the Name and leave the Units field blank.
   c. Click or tap Insert Expression and choose 1st Derivative(Y,X) as the expression.
   d. Select pH as Column Y and Volume as Column X. Click or tap Apply.
   e. To display a graph of d1 *vs*. volume, click or tap the y-axis label, select only d1, and dismiss the box.
   f. On the graph of d1 *vs*. volume, examine the data to determine the volume at the peak value of the first derivative.
2. Determine the zero value on the second derivative *vs*. volume plot.
   a. Click or tap More Options, ⋯, in the Volume column header in the table. Then, choose Add Calculated Column.
   b. Enter **d2** as the Name and leave the Units field blank.
   c. Click or tap Insert Expression and choose 2nd Derivative(Y,X) as the expression.
   d. Select pH as Column Y and Volume as Column X. Click or tap Apply.

e. Click or tap the y-axis label, select only d2 to display a graph of d1 *vs*. volume, and dismiss the box.

f. Click or tap the y-axis label, select only the d2 column, and dismiss the box. On the displayed graph of d2 *vs*. volume, examine the data to determine the volume when the 2nd derivative equals approximately zero.

PROCESSING THE DATA

1. Use your graph and data table to confirm the volume of NaOH titrant you recorded *before* and *after* the largest increase in pH values upon the addition of 1 drop of NaOH solution.

2. Determine the volume of NaOH added at the equivalence point. To do this, add the two NaOH values determined above and divide by two.

3. Calculate the number of moles of NaOH used.

4. See the equation for the neutralization reaction given in the introduction. Determine the number of moles of HCl used.

5. Recall that you pipeted out 10.0 mL of the unknown HCl solution for the titration. Calculate the HCl concentration.

DATA TABLE

Concentration of NaOH	M	M
NaOH volume added before largest pH increase	mL	mL
NaOH volume added after largest pH increase	mL	mL

Volume of NaOH added at equivalence point	mL	mL
Moles NaOH	mol	mol
Moles HCl	mol	mol
Concentration of HCl	mol/L	mol/L
Average [HCl]	M	