Acid-Base Titration

INTRODUCTION

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:

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

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 computer 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.

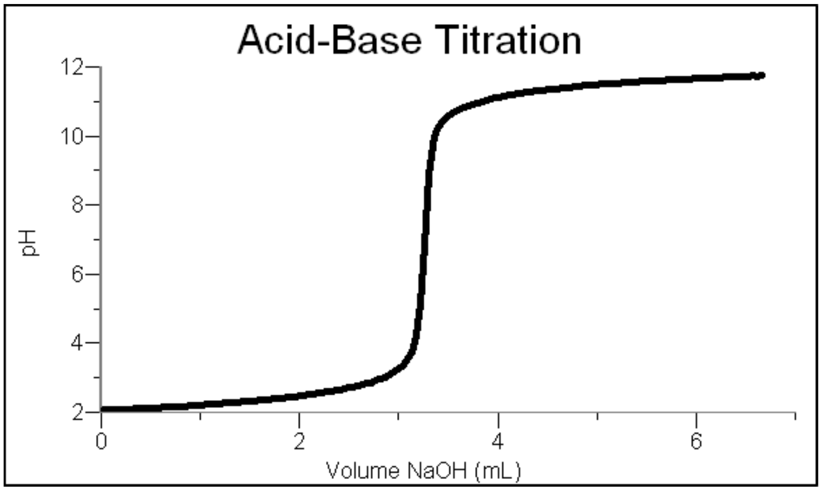


Figure 1

OBJECTIVES

In this experiment, you will

- 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.

Adapted from Experiment 22 in *Chemistry with Vernier*

MATERIALS

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

Computer
Vernier computer interface
Logger*Pro*
Vernier pH Sensor
HCl solution, unknown concentration
~0.1 M NaOH solution
Pipette bulb or pump
magnetic stirrer (if available)
stirring bar or Microstirrer (if available)
wash bottle
distilled water
ring stand
1 utility clamp
250 mL beaker

Materials required *only* for Method 1 (burette)

50 mL burette
10 mL pipette
2nd utility clamp
2nd 250 mL beaker

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

Vernier Drop Counter
60 mL reagent reservoir
5 mL pipette or graduated 10 mL pipette
100 mL beaker
10 mL graduated cylinder

CHOOSING A METHOD

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

Method 2 uses 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 Burette

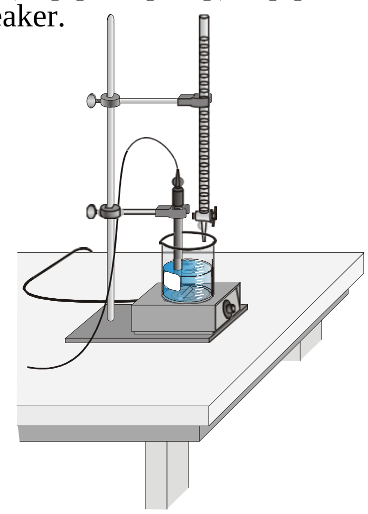
1. Obtain and wear goggles.

2. Add 50 mL of distilled water to a 250 mL beaker. Use a pipette bulb (or pipette pump) to pipette 10.0 mL of the HCl solution into the distilled water in the 250 mL beaker.

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 magnetic stirrer and add a stirring bar. If no magnetic stirrer is available, you need to stir with a stirring rod during the titration.

4. Use a utility clamp to suspend a pH Sensor on a ring stand as shown here. 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 magnetic stirrer, and adjust it to a medium stirring rate (with no splashing of solution).



Adapted from Experiment 22 in *Chemistry with Vernier*

5. Obtain approximately 60 mL of ~0.1 M NaOH solution in a 250 mL beaker. Obtain a 50 mL burette and rinse the burette with a few mL of the ~0.1 M NaOH solution. **WARNING**: *Sodium hydroxide solution,* NaOH: *Causes skin and eye irritation.*

   Use a utility clamp to attach the burette to the ring stand as shown here. Fill the burette a little above the 0.00 mL level of the burette with ~0.1 M NaOH solution. Drain a small amount of NaOH solution into the beaker so it fills the burette tip *and* leaves the NaOH at the 0.00 mL level of the burette. Record the precise concentration of the NaOH solution in your data table. Dispose of the waste solution from this step as directed by your teacher.

6. Connect the pH Sensor to the computer interface. Prepare the computer for data collection by opening the file "24a Acid-Base Titration" from the *Chemistry with Vernier* folder. Check to see that the pH value is between 2 and 3.

7. Before adding NaOH titrant, click Collect and monitor pH for 5-10 seconds. Once the displayed pH reading has stabilized, click Keep. In the edit box, type "0" (for 0 mL added). Press the ENTER key or click to store the first data pair for this experiment.

8. You are now ready to begin the titration. This process goes faster if one person manipulates and reads the burette while another person operates the computer and enters volumes.

   a. Add the next increment of NaOH titrant (enough to raise the pH about 0.15 units). When the pH stabilizes, again click Keep. In the edit box, type the current burette reading, to the nearest 0.01 mL. Press ENTER or click OK. You have now saved the second data pair.
   b. Continue adding NaOH solution in increments that raise the pH by about 0.15 units and enter the burette reading after each increment. Proceed in this manner until the pH is 3.5.
   c. When a pH value of approximately 3.5 is reached, change to a one-drop increment. Enter a new burette 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.
   d. 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 burette level after each increment.
   e. Continue adding NaOH solution until the pH value remains constant.

9. When you have finished collecting data, click Stop. Dispose of the beaker contents as directed by your teacher.

10. Print copies of the table and the graph.

11. If time permits, repeat the procedure.

METHOD 2: MEASURING VOLUME WITH A DROP COUNTER

1. Obtain and wear goggles and laboratory apron.
2. Connect the pH Sensor to CH 1 of the computer interface. Lower the Drop Counter onto a ring stand and connect its cable to DIG/SONIC 1.
3. Add 40 mL of distilled water to a 100 mL beaker. Use a pipette bulb (or pipette pump) to pipette 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.*

4. 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.*

5. 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 it with a few mL of the ~0.1 M NaOH solution. Use a utility clamp to attach the reagent reservoir to the ring stand. 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.

6. Prepare the computer for data collection by opening the file "24b Acid-Base (Drop Count)" from the *Chemistry with Vernier* folder.

7. To calibrate drops so that a precise volume of titrant is recorded in units of milliliters:
   a. From the Experiment menu, choose Calibrate ▸ DIG 1: Drop Counter (mL).
   b. Proceed by one of these two 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 OK. Then proceed directly to Step 8.
      - If you want to perform a new calibration, select the Automatic button, and continue with Step c below.
   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 (horizontal).
   e. Click the Start button.
   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 (read to the nearest 0.1 mL) in the edit box. Record the number of Drops / mL displayed on the screen for possible future use.
i. Click [OK]. Discard the NaOH solution in the graduated cylinder as indicated by your instructor and set the graduated cylinder aside.

8. 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, as shown in the small picture. 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 beaker containing the HCl solution onto the magnetic stirrer. Lower the pH Sensor into the beaker. Check to see that the pH value is between
   1.5 and 2.5.
   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.

9. Turn on the magnetic stirrer so that the Microstirrer is stirring at a fast rate.

10. You are now ready to begin collecting data. Click [▶ Collect]. 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.

11. 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 [■ Stop]. Turn the bottom valve of the reagent reservoir to a closed (horizontal) position.

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

13. Print copies of the table.

14. Print copies of the graph.

15. If time permits, repeat the procedure.

Adapted from Experiment 22 in *Chemistry with Vernier*

PROCESSING THE DATA

1. Use your graph and data table to determine the volume of NaOH titrant used in each trial. Examine the data to find the largest increase in pH values upon the addition of 1 drop of NaOH solution. Find and record the NaOH volume just *before* and *after* this jump.
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. Using the equation for the neutralization reaction given in the introduction, determine the number of moles of HCl used.
5. Calculate the HCl concentration using the volume of unknown HCl you pipetted out for each titration.
6. (Optional) If you did two titrations, determine the average [HCl] in mol/L.

DATA TABLE

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

Adapted from Experiment 22 in *Chemistry with Vernier*

EQUIVALENCE POINT DETERMINATION: Calculus 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. The equivalence point volume corresponds to the *peak* (maximum) value of the first derivative plot, and to the volume where the second derivative equals *zero* on the second derivative plot.

1. In Method 1, view the first-derivative graph (ΔpH/Δvol) by clicking the on the vertical-axis label (pH), and choose First Derivative. You may need to autoscale the new graph by clicking the Autoscale button, .

   In Method 2, you can also view the first derivative graph (pH/Δvol) on Page 2 of the experiment file by clicking the Next Page button, . On Page 2, you will see a plot of first derivative *vs.* volume.

2. In Method 1, view the second-derivative graph (Δ^2pH/Δvol^2) by clicking on the vertical-axis label, and choosing Second Derivative. In Method 2, view the second-derivative on Page 3 by clicking the Next Page button, .