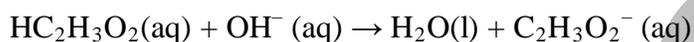
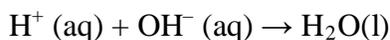


Acid-Base Titration

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 a base of known concentration. You will be testing a strong acid, HCl, solution and a weak acid, HC₂H₃O₂, solution. You will use the sodium hydroxide, NaOH, solution that you standardized in Lab 6 as your base of known concentration. The reaction equations are shown below in net ionic form.



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 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 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.

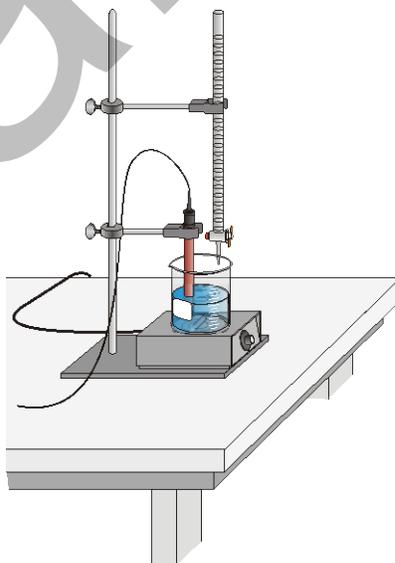


Figure 1

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	wash bottle
computer	distilled water
Vernier pH Sensor	ring stand
0.100 M sodium hydroxide, NaOH solution	utility clamp
hydrochloric acid, HCl, solution, unknown molarity	250 mL beaker
acetic acid, HC ₂ H ₃ O ₂ , solution, unknown molarity	10 mL pipet with pump
magnetic stirrer	50 mL graduated cylinder
stirring bar or Microstirrer	

Materials required *only* for Method 1 (buret)

50 mL buret	buret clamp
10 mL pipet	

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

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

METHOD 1: Measuring Volume Using a Buret

1. Obtain and wear goggles.
2. Obtain about 25 mL of a hydrochloric acid solution of unknown concentration. Add 50 mL of distilled water to a 250 mL beaker. Use a pipet bulb (or pipet pump) to transfer 10 mL of the HCl solution into the beaker. **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. Connect a pH Sensor to Channel 1 of a Vernier computer interface. Connect the interface to the computer using the proper cable.
5. Set up a ring stand, buret clamp, and 50.0 mL buret to conduct the titration (see Figure 1). Rinse and fill the buret with 0.100 M NaOH solution. **Note:** Record the concentration of the NaOH solution in your data table. **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, as shown in 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. Run the *Logger Pro* program on your computer. Open the file “07a Acid-Base” from the *Advanced Chemistry with Vernier* folder.
8. 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). Click to continue.
 - b. Add the next increment of NaOH (enough to raise the pH about 0.15 units). When the pH stabilizes, again click . In the edit box, type the current buret reading as accurately as possible. Click to continue.
 - c. 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 5.0 is reached, change to a one-drop increment.
 - d. After a pH value of approximately 10 is reached, add larger increments that raise the pH by about 0.15 pH units, and enter the buret level after each increment.
 - e. Continue adding NaOH solution until the pH value remains constant.
9. When you have finished collecting data, click . Dispose of the reaction mixture as directed. Rinse the pH Sensor with distilled water in preparation for the second titration.
10. 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 the Next Page button, .
 - b. Analyze the second derivative plot and record the volume of NaOH at the equivalence point.
11. Go back to the original titration graph. Conduct a second trial if directed by your instructor. If you wish to save the results of the titration, choose Store Latest Run from the Experiment menu.
12. Repeat the necessary steps to test the acetic acid solution. Conduct a second trial of the acetic acid solution if directed by your instructor. Analyze, print, and save the titration data for your acetic acid solution trial(s).

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. (You can add less, about 20 mL, if you will be using a stirring bar instead of the Microstirrer.) Use a pipet bulb (or pipet pump) to pipet 5.00 mL of the HCl solution into the 100 mL beaker with distilled water. **CAUTION:** *Handle the hydrochloric acid with care. It can cause painful burns if it comes in contact with the skin.*

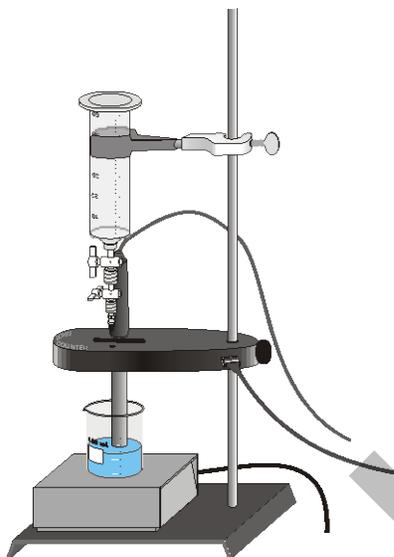


Figure 2

3. Lower the Drop Counter onto a ring stand and clamp it into position (see Figure 2).
4. Connect the Drop Counter to DIG/SONIC 1 of the Vernier computer interface. Connect the pH Sensor to CH 1 of the computer interface.
5. Run the *Logger Pro* program on your computer. Open the file “07b Acid-Base (Drop)” from the *Advanced Chemistry with Computers* folder.
6. 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 0.100 M NaOH solution and pour the rinse 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 0.100 M NaOH solution. **Note:** Record the precise concentration of the NaOH solution in your data table.
 - d. Place the 250 mL beaker, which contains the rinse NaOH, beneath the tip of the reservoir.
 - e. Drain a small amount of the NaOH solution into the 250 mL beaker so that 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.
7. 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 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 . Then proceed directly to Step 8.
 - 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. Keep the top valve closed.
 - e. Click Start.
 - 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 the NaOH solution in the graduated cylinder is between 9 and 10 mL, close the bottom valve of the reagent reservoir.
 - h. Enter the volume of NaOH (read to the nearest 0.1 mL) in the edit box.
 - i. Click . Discard the NaOH solution in the graduated cylinder as directed, 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. 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 HCl solution (from Step 2) 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.
 9. Turn on the magnetic stirrer so that the Microstirrer is stirring at a fast rate.
 10. You are now ready to conduct the titration. 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.
 11. Watch your graph to see when a large increase in pH takes place—this is the equivalence point of the reaction. When this jump in pH occurs, add about 3 more milliliters of NaOH solution, then click . Close the bottom valve of the reagent reservoir.
 12. Dispose of the beaker contents as directed.
 13. 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 examine 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 this plot to determine the volume of NaOH at the equivalence point.
 14. Return to the original titration graph. Conduct a second trial if directed by your instructor. To save the results of a titration, select Store Latest Run from the Experiment menu.

Computer 7

15. Repeat the titration with an acetic acid, $\text{HC}_2\text{H}_3\text{O}_2$, solution of unknown molar concentration. Conduct a second trial of the acetic acid solution if directed by your instructor.

DATA TABLE

HCl Trial	Volume HCl (mL)	[NaOH] (M)	Equivalence point (mL)
1			
2			

$\text{HC}_2\text{H}_3\text{O}_2$ Trial	Volume $\text{HC}_2\text{H}_3\text{O}_2$ (mL)	[NaOH] (M)	Equivalence point (mL)
1			
2			

DATA ANALYSIS

1. Calculate the molar amounts of NaOH used in the reaction with the HCl solution and with the $\text{HC}_2\text{H}_3\text{O}_2$ solution.
2. Calculate the molar concentration (molarity) of the HCl solution and the $\text{HC}_2\text{H}_3\text{O}_2$ 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.

TEACHER INFORMATION

Acid-Base Titration

1. The student pages with complete instructions for data collection using LabQuest App, Logger *Pro* (computers), EasyData (calculators), and a generic version can be found on the CD that accompanies this book. See *Appendix A* for more information.
2. Prepare the 0.1 M HCl solution by adding 8.6 mL of concentrated HCl to sufficient distilled water to make 1 L of solution.
3. Prepare the 0.1 M HC₂H₃O₂ solution by adding 5.7 mL of glacial acetic acid to sufficient water to make 1 L of solution.
4. For best results, use a standardized solution of ~0.1 M NaOH (e.g., 0.0941 M).
5. The stored calibration information for the pH Sensor in the data-collection programs (LabQuest App, Logger *Pro*, or EasyData) works well for this experiment.
6. It is possible to zoom in on the equivalence point in the 2nd derivative vs. volume plot, and then perform a linear fit on the data near the equivalence point. Once a linear fit is established, it is possible to interpolate along that fit until the 2nd derivative value is approximately 0, corresponding to the equivalence point volume of NaOH titrant (see Sample Graphs on the following pages).
7. If your students are using the Drop Counter, note that the volumes of reagents are reduced.
8. For best results, instruct your students to perform a new calibration on their Drop Counters. If time does not permit a new calibration, the calibration of 28 drops/mL, stored in the Logger *Pro* or the LabQuest App, may be used. This value will provide good results.
9. If you are using a calculator and EasyData, there are several different combinations of equipment that will work for collecting pH data. The most common method, which works for both the TI-83 Plus and TI-84 Plus families of calculators, is to use a pH Sensor connected to a LabPro or a CBL 2. This is the equipment that must be used if you are collecting data with a calculator and a Drop Counter (Method 2 of the Procedure).

The other combination of equipment, which uses the USB port on a TI-84 Plus calculator, is to collect data with a pH Sensor connected to an EasyLink. The EasyLink cannot be used with a Drop Counter because the Drop Counter is not supported by the EasyLink.

HAZARD ALERTS

Sodium hydroxide: Corrosive solid; skin burns are possible; much heat evolves when added to water; very dangerous to eyes; wear face and eye protection when using this substance. Wear gloves. Hazard code: B—Hazardous.

Hydrochloric acid: Highly toxic by ingestion or inhalation; severely corrosive to skin and eyes. Hazard code: A—Extremely hazardous.

Experiment 7

Acetic acid: Corrosive to skin and tissue; moderate fire risk (flash point: 39°C); moderately toxic by ingestion and inhalation. Hazard Code: A—Extremely hazardous.

The hazard information reference is: Flinn Scientific, Inc., *Chemical and Biological Catalog Reference Manual*, P.O. Box 219, Batavia, IL 60510, (800) 452-1261, www.flinnsci.com.

SAMPLE DATA

HCl Trial	Volume HCl (mL)	[NaOH] (M)	Equivalence point (mL)	HC ₂ H ₃ O ₂ Trial	Volume HC ₂ H ₃ O ₂ (mL)	[NaOH] (M)	Equivalence point (mL)
1	5.00	0.0941	5.55	1	5.00	0.0941	5.64
2	5.00	0.0941	5.53	2	5.00	0.0941	5.66

Note: The sample data were collected by conducting the titration with a Drop Counter.

ANSWERS TO THE DATA ANALYSIS QUESTIONS

- Answers will vary. For the sample HCl data (Trial 2), 5.20×10^{-4} mol of 0.0941 M NaOH (5.53 mL) neutralized 5.00 mL of HCl solution. For the sample HC₂H₃O₂ data (Trial 2), 5.33×10^{-4} mol of 0.0941 M NaOH (5.66 mL) neutralized 5.00 mL of HC₂H₃O₂ solution.
- Answers will vary. In the sample HCl data (Trial 2), the molarity is calculated as shown here.

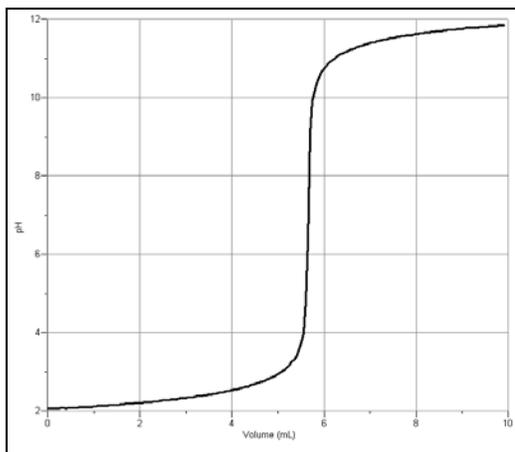
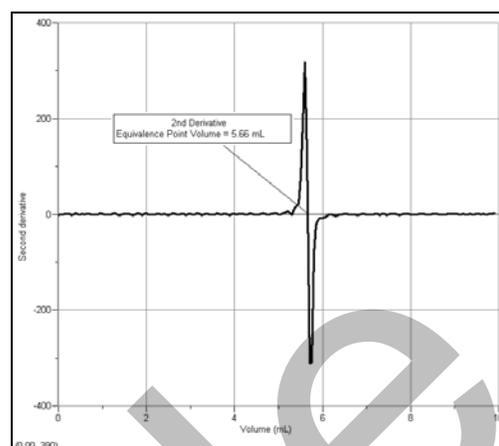
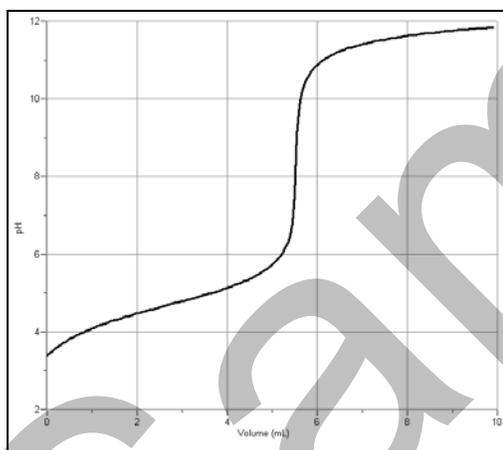
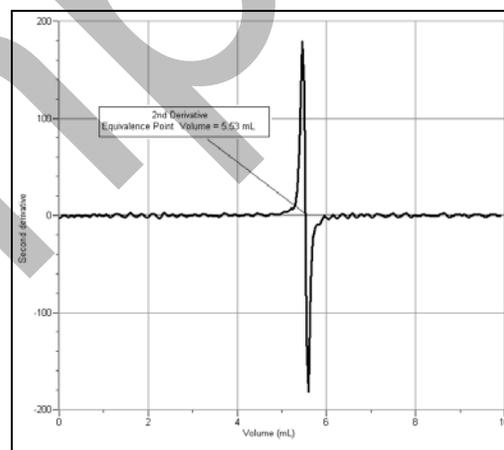
$$5.20 \times 10^{-4} \text{ mol} \div 0.00500 \text{ L} = 0.104 \text{ M.}$$

In the sample HC₂H₃O₂ data (Trial 2), the molarity is calculated as shown here.

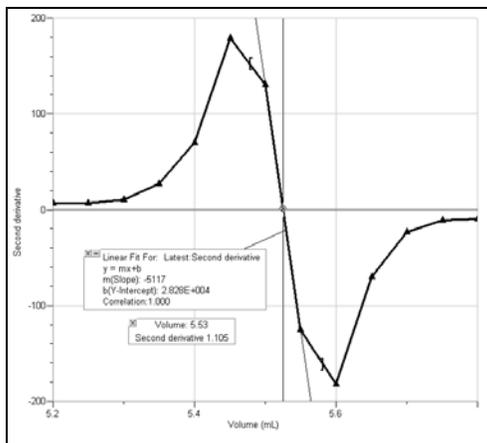
$$5.33 \times 10^{-4} \text{ mol} \div 0.00500 \text{ L} = 0.107 \text{ M.}$$

- For the sample data, both acid solution molarities compared well with the actual molarities.
- Answers will vary. A complete answer will refer to the conjugate base formed in the weak acid – strong base titration that affects the pH of the equivalence point.

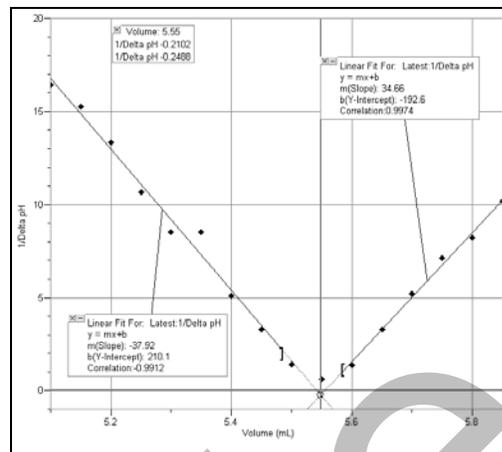
SAMPLE GRAPHS

*pH vs. volume of HCl solution**2nd derivative vs. volume of HCl solution**pH vs. volume of acetic acid**2nd derivative vs. volume of acetic acid*

Experiment 7



Interpolate linear fit of 2nd derivative to zero



Gran plot (1/delta pH vs. volume)