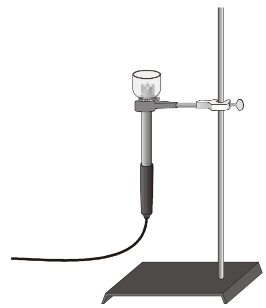
PROCEDURE

1. Obtain and wear goggles.

2. Prepare the pH Sensor for data collection.
   a. Connect the pH Sensor to LabQuest and choose New from the File menu. If you have an older sensor that does not auto-ID, manually set up the sensor.
   b. Remove the pH Sensor from the pH storage solution bottle by unscrewing the lid. Carefully slide the lid from the sensor body.
   c. Rinse the tip of the sensor with distilled water.
   d. Obtain a pH Sensor storage bottle that has been cut in half. This is your microbeaker!
   e. With the open end of the pH Sensor pointing upward, as shown here, slip the microbeaker and cap down onto the sensor body (small opening first), so the sensor tip extends about 1 cm into the bowl of the microbeaker. Then tighten the threads of the cap so the cap tightens snugly against the pH Sensor body.
   f. Attach the utility clamp to a ring stand and to the bottle lid, with the sensor in an inverted position as shown here.



3. Obtain a dropper bottle containing the HCl solution of unknown concentration. Add 10 drops of the HCl solution into the micro-beaker. As you add the drops, hold the bottle in a vertical position to ensure that drop size is uniform. **CAUTION:** *Handle the hydrochloric acid with care. It can cause painful burns if it comes in contact with the skin.* Add 1 drop of phenolphthalein indicator to the microbeaker, then add enough distilled water so the resulting solution completely covers the sensor tip. Stir the solution thoroughly with the toothpick.

4. Obtain a dropper bottle containing 0.10 M NaOH. Wait until Step 6 to begin adding this solution to the HCl solution in the microbeaker.

5. Set up the data-collection mode.
   a. On the Meter screen, tap Mode. Change the data-collection mode to Events with Entry.
   b. Enter the Name (Volume) and Units (drops). Select OK.

6. You are now ready to perform the titration. This process goes faster if one person adds drops of NaOH solution while another person enters volumes.
   a. Start data collection.
   b. Before you have added any drops of NaOH solution, tap Keep and enter **0** as the NaOH volume, in drops. Select OK to store the first data pair for this experiment.
   c. Add one drop of NaOH solution. Be sure to hold the dropper bottle vertically to ensure that the drop size is uniform. **CAUTION:** *Sodium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.* Stir with a toothpick to uniformly mix the solution. When the pH stabilizes, tap Keep and enter **1** as the number of drops of NaOH solution added. Select OK. You have now saved the second data pair for the experiment.
   d. Add a second drop of NaOH solution, stir. When the pH stabilizes, tap Keep and enter **2** as the number of drops of NaOH solution added. Select OK.
   e. Continue this procedure until 20 drops of NaOH solution have been added.

7. Stop data collection.

8. Examine the data on the graph of pH *vs.* drops to find the *equivalence point*—that is, the 1 drop volume increment that resulted in the largest increase in pH. As you tap each data point (or use the ► or ◄ keys on LabQuest), the pH and volume values are displayed. Go to the region of the graph with the large increase in pH. Find the NaOH volume (in drops) 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.

9. Carefully loosen the pH Sensor from the utility clamp and dispose of the beaker contents as directed by your instructor. Thoroughly rinse out the microbeaker and the pH Sensor tip. Remove the microbeaker from the sensor. Rinse the sensor tip with distilled water and return it to the pH Sensor storage bottle.

10. Print a copy of the graph of pH *vs*. volume. Then print a copy of the NaOH volume data and the pH data for the titration.

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. Tap the Table tab. Choose New Calculated Column from the Table menu.
   b. Enter d1 as the Calculated Column Name. Select the equation, 1st derivative (Y,X). Use Volume as the Column for X, and pH as the Column for Y. Select OK.
   c. On the displayed plot of d1 *vs.* volume, examine the graph 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. Tap the Table tab. Choose New Calculated Column from the Table menu.
   b. Enter d2 as the Calculated Column Name. Select the equation, 2nd derivative (Y,X). Use Volume as the Column for X, and pH as the Column for Y. Select OK.
   c. On the displayed plot of d2 *vs.* volume, examine the graph to determine the volume when the 2nd derivative equals approximately zero.

PROCESSING THE DATA

1. Use your printed graph 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 (use 0.5 drop increments in your answer).

3. Using the formula in the introduction of the experiment, calculate the concentration of the hydrochloric acid solution (in M or mol/L).

DATA AND CALULATIONS TABLE

Concentration of NaOH	M
NaOH volume added *before* the largest pH increase	drops
NaOH volume added *after* the largest pH increase	drops
Volume of NaOH added at equivalence point	drops
Concentration of HCl	M

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.
   d. Tap the Table tab. Choose New Calculated Column from the Table menu.
   e. Enter d1 as the Calculated Column Name. Select the equation, 1st derivative (Y,X). Use Volume as the Column for X, and pH as the Column for Y. Select OK.
   f. On the displayed plot of d1 *vs.* volume, examine the graph to determine the volume at the peak value of the first derivative.
2. Determine the zero value on the second derivative *vs.* volume plot.
   g. Tap the Table tab. Choose New Calculated Column from the Table menu.
   h. Enter d2 as the Calculated Column Name. Select the equation, 2nd derivative (Y,X). Use Volume as the Column for X, and pH as the Column for Y. Select OK.
   i. On the displayed plot of d2 *vs.* volume, examine the graph to determine the volume when the 2nd derivative equals approximately zero.

TEACHER INFORMATION

Microscale Acid-Base Titration

1. The student pages with complete instructions for data-collection using LabQuest App, Logger *Pro* (computers), EasyData or DataMate (calculators), and DataPro (Palm handhelds) can be found on the CD that accompanies this book. See *Appendix A* for more information.

2. This experiment can be done prior to Experiment 24, “Acid-Base Titration”. Students will quickly discover the shape of an acid-base titration curve for the reaction between a strong acid and strong base. They should not expect to determine precise concentration values using this method. It is meant to be an introduction to a traditional acid-base titration, not a substitute for more precise methods.

3. You can purchase additional pH storage solution bottles from Vernier to use as the microbeakers. Simply cut the bottles in half to use in this experiment. Distribute the half with the threaded opening to student lab stations. You do not need to include the cap, since students are instructed to use the cap and O-ring already on the probe). Order information is:

   pH Storage Solution Bottles pkg of 5 order code: BTL

4. Explain to your students the purpose of adding the phenolphthalein indicator. They can easily observe the color change and large pH increase occur simultaneously in this experiment.

5. The preparation of 0.10 M NaOH requires 4.0 g of NaOH per liter of solution. Since the equivalence point concentrations are only approximate, using a value of ~0.10 M works well for this experiment. **HAZARD ALERT:** 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.

6. Unknown samples with HCl concentrations in the 0.080 to 0.100 M range work well. The preparation of 0.080 M HCl requires 6.7 mL of concentrated HCl per liter of solution. HCl that is 0.100 M requires 8.4 mL of concentrated reagent per liter. **HAZARD ALERT:** Highly toxic by ingestion or inhalation; severely corrosive to skin and eyes. Hazard Code: A—Extremely hazardous.

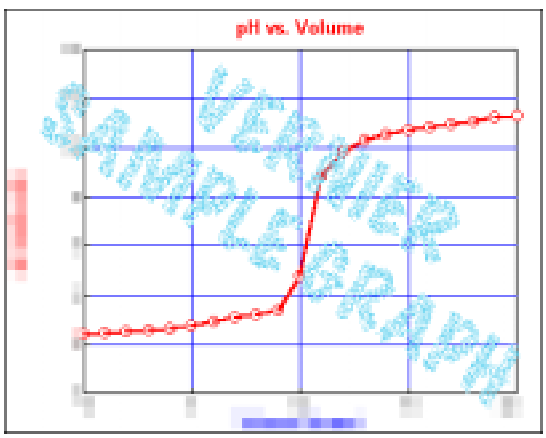
   The hazard information reference is: Flinn Scientific, Inc., *Chemical & Biological Catalog Reference Manual*, 1-800-452-1261, www.flinnsci.com. See *Appendix D,* of this book, *Chemistry with Vernier*, for more information.

7. The HCl and NaOH solutions can be dispensed from microscale Beral pipets if you do not have dropper bottles.

8. The stored pH calibration works well for this experiment.

SAMPLE RESULTS

Concentration of NaOH	XXXX
NaOH volume added before largest pH increase	XXXX
NaOH volume added after largest pH increase	XXXX
Volume of NaOH added at equivalence point	XXXX
Concentration of HCl	XXXX



Microscale titration for sodium hydroxide and hydrochloric acid