Experiment
31

Microscale 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:

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

When HCl solution is titrated with NaOH solution, the pH value 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. The change in pH then becomes more gradual again, before leveling off with the addition of excess base.

Since this experiment may be your introduction to acid-base titrations, you will determine only the *approximate* concentration of the hydrochloric acid solution. Use the formula:

$$M_{\text{acid}} = M_{\text{base}} \times \frac{V_{\text{base}}}{V_{\text{acid}}}$$

where M_{acid} is the concentration of the acid (in M or mol/L), M_{base} is the concentration of the base, V_{base} is the volume of the base (in drops), and V_{acid} is the volume of the acid. The concentration of the sodium hydroxide solution is 0.10 M. The drops of sodium hydroxide and hydrochloric acid solutions at the equivalence point will be determined from the experiment.

OBJECTIVES

In this experiment, you will

- Perform a microscale acid-base titration.
- Monitor pH.
- Determine the approximate concentration of the acid used in the titration.

MATERIALS

computer
Vernier computer interface
Logger*Pro*
Vernier pH Sensor
wash bottle
distilled water
toothpick (for stirring)
0.10 M NaOH solution (in a dropper bottle)
HCl solution (in a dropper bottle)
ring stand
utility clamp
phenolphthalein indicator
micro-beaker (top half of a storage bottle for the pH Sensor)

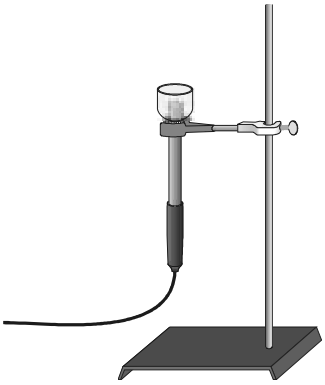
PROCEDURE

1. Obtain and wear goggles.

2. Connect the pH Sensor to the computer interface. Prepare the computer for data collection by opening the file "31 Microscale Titration" from the *Chemistry with Computers* folder of Logger*Pro*.

3. Prepare the pH Sensor for data collection.

   a. Remove the pH Sensor from the pH storage solution bottle by unscrewing the lid. Carefully slide the lid from the sensor body.
   b. Rinse the tip of the sensor with distilled water.
   c. Obtain a pH Sensor storage bottle that has been cut in half. This is your microbeaker!
   d. 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.
   e. Attach the utility clamp to a ring stand and to the bottle lid, with the sensor in an inverted position as shown here.



4. 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.

5. Obtain a dropper bottle containing 0.10 M NaOH. Wait until Step 7 to begin adding this solution to the HCl solution in the microbeaker.

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

7. You are now ready to begin the titration. This process goes faster if one person adds the NaOH solution and stirs, while another person operates the computer and enters the number of drops.

   a. Add one drop of NaOH titrant to the micro-beaker. 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, again click Keep. In the edit box, type "1" for the number of drops of NaOH solution added. Press ENTER. You have now saved the second data pair for the experiment.
   b. Add a second drop of NaOH solution, stir, and click Keep when the pH stabilizes. Enter "2" for the number of drops added.
   c. Continue this procedure until 20 drops of NaOH solution have been added.

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

9. Print a copy of the graph. Enter your name(s) and the number of copies you want to print.

10. Print a copy of the table. Enter your name(s) and the number of copies you want to print.

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 CALCULATIONS 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

TEACHER INFORMATION

Microscale Acid-Base Titration

1. 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.

2. 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

3. 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.

4. 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.

5. 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 Computers*, for more information.

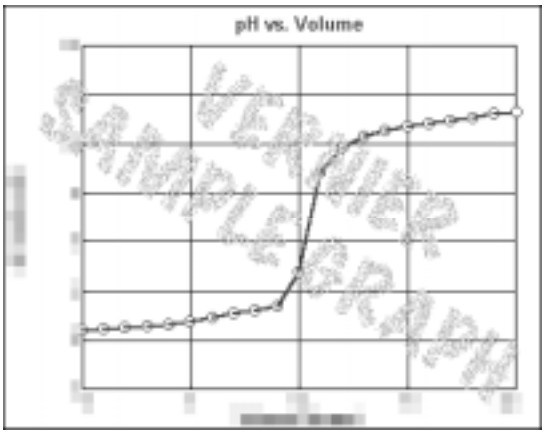
6. The HCl and NaOH solutions can be dispensed from microscale Beral pipets if you do not have dropper bottles.

7. 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