Experiment 7
Titration Curves of Strong and Weak Acids and Bases

Goals

To calibrate a pH probe. To become familiar with acid-base titration curves. To determine the concentration of an unknown acid.

Equipment and Materials

0.10 M ammonium hydroxide solution, 0.10 M sodium hydroxide solution, 0.0074 M hydrochloric acid solution, 0.0074 M acetic acid solution, a solution of hydrochloric acid with an unknown concentration, phenolphthalein solution, magnetic stir plate with magnetic stirrer, 50 mL burettes (2), 100 mL graduated cylinder

Discussion

When a base is added to an acid there will be a change in the pH of the solution. The pH depends on the amount of acid, base, and the nature of the acid and the base. Strong acids and bases have a greater effect on pH than weak acids and weak bases at the same concentration. Examples of acid-base reactions include:

$$\underset{\text{strong acid}}{HCl(aq)} + \underset{\text{strong base}}{NaOH(aq)} \rightarrow H_2O(l) + NaCl(aq)$$

$$\underset{\text{weak acid}}{HC_2H_3O_2(aq)} + \underset{\text{strong base}}{NaOH(aq)} \rightarrow NaC_2H_3O_2(aq) + H_2O(l)$$

$$\underset{\text{strong acid}}{HCl(aq)} + \underset{\text{weak base}}{NH_3(aq)} \rightarrow NH_4^+(aq) + Cl^-(aq)$$

$$\underset{\text{weak acid}}{HC_2H_3O_2(aq)} + \underset{\text{weak base}}{NH_3(aq)} \rightarrow NH_4^+(aq) + C_2H_3O_2^-(aq)$$

In this experiment we will look at the titration curves that result from the four reactions written above. We will then take advantage of the point of inflection of a titration curve to determine the molarity of a sample of hydrochloric acid solution.

Procedure

SAFETY PRECAUTIONS
Safety glasses are required for this experiment. Hydrochloric acid can burn skin and should be handled with care. Sodium hydroxide can damage skin and should be handled with care. The glass electrode is a fragile device and should be handled with care.

Students work in pairs throughout this experiment.

Part 1 Calibration of pH glass electrode

1. The calibration of the pH glass electrode requires the measurement of standard solutions at two different pH values. Pick two buffers to use for the calibration and obtain about 10 mL of each in small glass vials.

2. Connect the LoggerPro data interface and pH glass electrode.

3. Start the computer application Logger Pro 3.3 and check to see that a pH value is displayed on the computer monitor. If no value is displayed, check all connections and re-start the application.

4. The glass electrode can be taken from its storage solution and rinsed with distilled or deionized water. The glass electrode should never be exposed to air for more than a few seconds.

5. Place the electrode in the first buffer solution.

6. Open the **Calibrate** function of LoggerPro from the **Experiment** menu.

7. The window should have **Live Calibration** selected. Select **Calibrate Now**.

8. Gently stir the buffer solution with the pH probe for 5-10 seconds. Once the displayed voltage stabilizes record the pH of the buffer solution in the window and in the data sheet. Record the voltage reading displayed in the window in the data sheet and select **Keep**.

9. Repeat the same procedure for the second standard solution. The glass electrode should always be rinsed with distilled or deionized water when transferring it to a new solution.

Part 2. Shapes of acid-base titration curves

Four titration curves will be plotted on the same chart. By plotting the titration curves together we can note the similarities and differences between weak and strong acids and bases. The shapes of these four curves will be compared and contrasted. In each case the basic solution will be added to the acidic solution. The four titrations are:

sodium hydroxide + hydrochloric acid	ammonium hydroxide + hydrochloric acid
sodium hydroxide + acetic acid	ammonium hydroxide + acetic acid

1. Prepare the LoggerPro software to plot data for titration curves. Use the Data **collection** option in the **Experiment** menu to set the experiment for an experiment length of 500 seconds and a data sampling rate of 0.5 samples per second.

2. Rescale the y-axis to a pH range of 2-12.

3. Clean two 50.00 mL burettes using tap water and then distilled or deionized water.

4. Rinse one of the burettes with a few milliliters of 0.10 M sodium hydroxide solution. Fill the burette to the 0.00 mL mark with the sodium hydroxide solution.

5. Rinse the second burette with a few milliliters of 0.10 M ammonium hydroxide solution. Fill the burette to the 0.00 mL mark with the ammonium hydroxide solution.

6. Using a graduated cylinder add 100 mL of 0.0074 M hydrochloric acid solution to a clean and dry 250 mL beaker. Add two drops of phenolphthalein indicator to the beaker. Place a magnetic stir bar to the solution and place the beaker onto a magnetic stirrer. Turn on the stirrer so that the solution is stirred without splashing onto the sides of the beaker.

7. Place the pH probe into the solution and clamp the probe to the ring stand. The pH should be near 2.

8. Begin collecting data. After thirty seconds of data collection, open the valve to the burette and allow a constant flow of the basic solution to drop into the acidic solution. The flow rate should be near one drop per second.

9. Monitor the beaker as the base is added to the acid. Record the time at which the color of the solution changes from clear to a faint pink.

9. At the end of the 500 second run select **Store Latest Run** from the **Experiment** menu. Label the curve using the **Text** option in the **Insert** menu.

10. Repeat the above steps for the three remaining titrations. Each titration curve should be labeled immediately following the titration using **text annotation** from the **Experiment** menu. All four titrations should appear on the same chart.

11. Be sure that the chart is titled.

Part 3 Quantitative Acid-base Titration

1. Obtain and wear eye protection. You will work in groups as assigned by your instructor.

2. Use a Pipet Pump and the 25-mL volumetric pipet to add 25.00 mL of the HCl solution into a 250-mL beaker. Enter this volume in the Data and Results Table. **CAUTION:** Handle the hydrochloric acid with care. It can cause painful burns if it comes in contact with the skin.

3. Add 100 mL of distilled water to the beaker. Place a plastic coated magnet in the beaker.

4. Use a utility clamp to suspend a pH sensor on a ring stand. Position the pH Sensor in the HCl solution and adjust its position at one side of the beaker so that its end is about ¼" above the plastic coated magnet.

5. Use a buret reader to taking volume readings. Your instructor will show you how to use a buret reader.

6. Rinse the 50-mL buret with a 3–5 milliliters of the ~0.1 M NaOH solution. Use a buret clamp to attach the buret to the ring stand. 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 into a waste beaker so it fills the buret tip and leaves the NaOH at the 0.00-mL level of the buret. Examine the tip of the buret carefully to be sure that there are no air bubbles in it. If there are, let the NaOH solution drip into the waste beaker as fast as possible until you have swept out the bubbles. Refill the buret and re-adjust the volume to the 0.00-mL mark. Record the precise concentration of the NaOH solution in your data table. Dispose of the waste solution into the sink.

CAUTION**:** Sodium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.

7. Prepare the computer for data collection by opening the file in the Experiment 24a folder of Chemistry with Computers. The vertical axis has pH scaled from 0 to 14 pH units. The horizontal axis has volume scaled from 0 to 25 mL. Check to see that the Meter window shows a pH value between 2 and 3. You should rescale the pH axis so it runs from a pH of 1 to a pH of 12, and rescale the volume axis so it runs from 0 mL to 40 mL.

Also check to be sure that the pH and the volume can be entered to 2 decimal places, and that the first derivative is calculated to 3 decimal places. In the Data menu select Columns Options and then Volume. Set the Displayed Precision to 2 decimal places. Click OK. Repeat: Data → Columns Options → pH and set the Displayed Precision to 2 decimal places. Repeat: Data → Columns Options → d1 and set the Displayed Precision to 3 decimal places. (d1 is the abbreviation for the 1st derivative, and d2 is the abbreviation for the 2nd derivative.)

8. Before adding NaOH titrant, start the magnetic stirrer, click Collect and monitor the pH for 5-10 seconds. Once the displayed pH reading has stopped changing, click Keep. In the Events with Entry box, type "0.00" (for 0.00-mL added). Press the ENTER key to store the first data pair for this experiment.

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

a. Add the first increment of NaOH titrant—enough to raise the pH about 0.2 units. When the pH stabilizes, again click Keep. In the Events with Entry box, type the current buret volume reading, to the nearest 0.01 mL. Press ENTER.

b. Continue adding NaOH solution in increments that raise the pH by about 0.2 units and enter the buret volume reading after each increment.

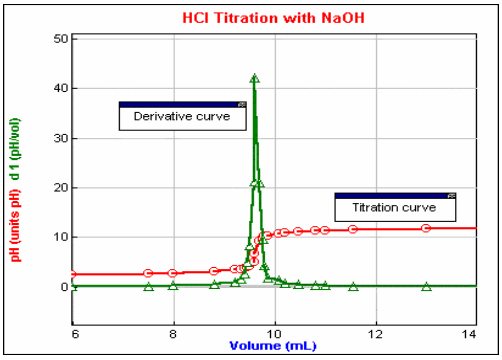
When a pH value of approximately 3.5 is reached, change to a one-drop increment. Enter a new buret volume reading after each drop is added. Note: To get the best-looking titration curve, each increment should be the same size—namely one drop.

c. After a pH value of approximately 10.5 is reached, again add larger increments of NaOH that raise the pH by about 0.2 pH units, and enter the buret volume reading after each increment.

d. Continue adding NaOH solution until you have added about 40 mL of titrant in total.

10. When you have finished collecting data, click Stop. Dispose of the beaker contents into the sink.

11. To delete a "bad" data point, select it and in the Edit menu choose Strike Through Rows.
A note about the first derivative: The first derivative gives the value of the slope of the titration curve at any point along the curve. You can see that at the left of the titration curve, the slope is small. In the vertical region of the curve, the slope is large. And at the right of the curve, the slope is again small. That's why the derivative curve is first small, then has a large spike, and then is small again.



12. Now you will display the first derivative of the titration curve and the titration curve on the same graph. Click on the pH axis label and check d1 in the dialog box. Click OK. The graph will now show both the pH curve and the first derivative curve. Rescale the graph by clicking Autoscale Once in the View menu.
Remove the point protectors by double clicking anywhere on the graph to make the graph the active window. In the Graph Options box, uncheck Point Protectors to reduce the clutter on the graph. Annotate each curve as shown above. If you wish, save your data to a floppy disk as described in Experiment I.

13. Print as many copies of the Data Table as there are people in your group.

14. Print the graph in Landscape mode.

Data Analysis
1. Look at the Table Window. Ignore the column labeled d2. (This column is the second derivative—it has a value of zero at the inflection point.) In the column labeled d1, find the largest number. The corresponding volume and pH are at the inflection point of the titration curve. [On your graph, notice that the peak of the d1 curve occurs at the inflection point of the titration curve.] This volume is the equivalent volume of NaOH at the equivalence point.

2. From the volume of NaOH determined in step 1 above, calculate the number of moles of NaOH used.

$$M_{NaOH}\, V_{NaOH} = \text{moles NaOH}$$

3. Refer to the equation for the neutralization reaction given in the introduction. Determine the number of moles of HCl used: HCl(aq) + NaOH(aq) → H_2O(l) + NaCl(aq)

4. Using the volume of the HCl solution that was pipetted into the beaker, and the number of moles of HCl calculated in #3 above, calculate the molarity of the HCl solution.

$$M_{HCl} = \frac{\text{moles HCl}}{V_{HCl}}$$

Data Sheet

Experiment 7: Titration Curves of Strong and Weak Acids and Bases

Part 1. Calibration of pH glass electrode

pH of first buffer solution _____ voltage _____

pH of second buffer solution _____ voltage _____

Part 2. Shapes of acid-base titration curves

Titration	**initial pH**	**final pH**	**time of color change**	**pH at color change**
HCl + NaOH	_____	_____	_____	_____
HAc + NaOH	_____	_____	_____	_____
HCl + NH_4OH	_____	_____	_____	_____
HAc + NH_4OH	_____	_____	_____	_____

Part 3. Quantitative Determination of the Concentration of HCl

Volume of HCl added to beaker	mL	mL
Concentration of NaOH (obtained from your instructor)	M	M
Volume of NaOH added at the equivalence point	mL	mL
Moles of NaOH added at the equivalence point	mol	mol
Moles of HCl in the beaker	mol	mol
Calculated concentration of HCl (reported to 4 significant figures)	M	M
For the instructor's use only	% error =	Point deficit =

Acid Base Indicators

Indicator	pH range	color change	Preparation
methyl violet	0.0 - 1.6	yellow to blue	< 0.1% in water
crystal violet	0.0 - 1.8	yellow to blue	< 0.1% in water
malachite green	0.2 - 1.8	yellow to blue-green	0.1% in water
erythrosine, disodium	2.2 -3.6	orange to red	0.1% in water
bromophenol blue	3.0 - 4.6	yellow to blue	basic solutions
congo red	3.0 -5.0	blue to red	0.1% in water
methyl orange	3.2 -4.4	red to yellow	
ethyl orange	3.4 - 4.8	red to yellow	
ethyl red	4.0 - 5.8	colorless to red	
bromocresol green	4.0 - 5.4	yellow to blue	
alizarin red S	4.6 - 6.0	yellow to red	
methyl red	4.8 - 6.0	red to yellow	
p-nitrophenol	5.4 - 6.6	colorless to yellow	
alizarin	5.6 - 7.2	yellow to red	
	11.0 - 12.4	yellow to red	
brilliant yellow	6.6 - 7.8	yellow to orange	
phenol red	6.6 - 8.0	yellow to red	
cresol red	7.0 - 8.8	red to yellow	
thymol blue	8.0 - 9.6	red to yellow	
phenolphthalein	8.2 - 10.0	colorless to pink	
thymolphthalein	9.4 - 10.6	colorless to blue	
alizarin yellow R	10.1 - 12.0	yellow to red	
2,4,6-trinitrotoluene	11.5 - 13.0	colorless to orange	