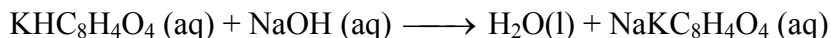
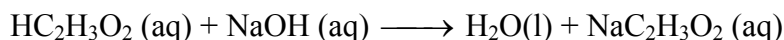


Acid-Base Titration

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 perform 2 titrations; one using potassium acid phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$) (KHP) with a basic sodium hydroxide solution, NaOH. The concentration of the NaOH solution will be determined. Hydrogen ions from the KHP react with hydroxide ions from the NaOH in a one-to-one ratio to produce water in the overall reaction:



In the second part of the experiment, an unknown acetic acid ($\text{HC}_2\text{H}_3\text{O}_2$) solution will be titrated with the NaOH solution from the first part of the experiment. This is also a one-to-one ratio reaction:



When a KHP 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. This is also the case with an acetic acid solution being titrated with NaOH. The procedure for Part 1 and 2 of this experiment is basically the same; just different acids in each case. The first part is done to find the concentration of the NaOH solution and the second part is done to determine the concentration of the unknown.

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.

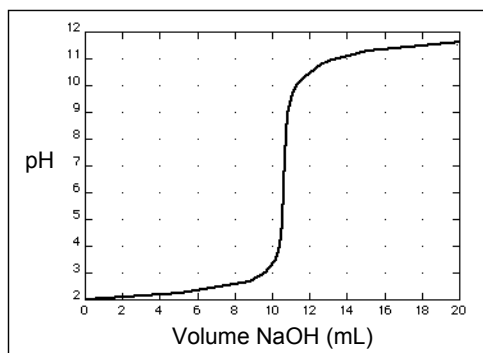


Figure 1

OBJECTIVES

In this experiment, you will

Experiment 24

- Use a pH Sensor to monitor changes in pH as sodium hydroxide solution is added to the acid solutions
- 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.

Two methods are described below. we will be using only **Method 1**.

MATERIALS

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

computer	magnetic stirrer (if available)
Vernier computer interface	stirring bar or Microstirrer (if available)
LoggerPro	wash bottle
Vernier pH Sensor	distilled water
Acetic acid solution of unknown concentration	ring stand
50% by weight NaOH solution	1 utility clamp
pipet bulb or pump	250 mL beaker

Materials required *only* for Method 1 (buret)

50 mL buret	2nd utility clamp
5 mL pipet	2nd 250 mL beaker

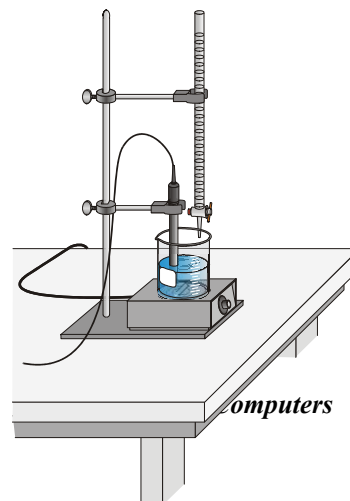
CHOOSING A METHOD

Method 1 has the student deliver volumes of NaOH titrant from a buret. After titrant is added, and pH values have stabilized, the student is prompted to enter the buret 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 Buret

1. Obtain and wear goggles.
2. Add 50 mL of distilled water to a 250 mL beaker. Accurately weigh between 0.600g and 0.700 g of KHP, recording the exact mass on the data sheet. Add the KHP to the beaker of water
3. Place the beaker on a magnetic stirrer and add a stirring bar.
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).



5. Prepare your NaOH solution by adding approximately 250 mL of distilled water to a 500 mL Florence flask. Then add about 3 mL of a 50% by mass NaOH solution to the water.
CAUTION: *Sodium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.* Rinse out the graduated cylinder with a couple of mL of distilled water twice and add the rinsings to the Florence flask and mix well. You will use this solution for all the titrations in today's experiment.
6. Obtain a 50 mL buret and rinse the buret with a few mL of the NaOH solution. Use a utility clamp to attach the buret to the ring stand as shown here. Fill the buret a little above the 0.00 mL level of the buret with the 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. Dispose of the waste solution from this step as directed by your teacher.
7. 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 Computers* folder. Check to see that the pH value is between 2 and 3.
7. Before adding NaOH titrant, click and monitor pH for 5-10 seconds. Once the displayed pH reading has stabilized, click . 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 buret 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 . In the edit box, type the current buret reading, to the nearest 0.01 mL. Press ENTER or click . You have now saved the second data pair for the experiment.
 - b. Continue adding NaOH solution in increments that raise the pH by about 0.15 units and enter the buret reading after each increment. Proceed in this manner until the pH is between 3.5 and 4.0.
 - c. When a pH value of approximately 3.5 is reached, change to a one-drop increment. Enter a new buret 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. **A convenient way to do this is to turn the stopcock 180° quickly and smoothly.**
 - 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 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 beaker contents as directed by your teacher.
10. Print copies of the table and the graph.
11. If time permits, repeat the procedure.
12. For Part 2, Obtain a 5.00 mL volumetric pipet, clean it thoroughly, rinsing finally with tap water (twice), distilled water (once) and twice with your unknown sample. **Be sure to record your unknown #.** Using this pipet, carefully and accurately measure 5.00 mL of your unknownr sample into two clean 250 mL Erlenmeyer flasks. The instructor will illustrate proper pipetting technique. Titrate each with the standardized NaOH solution, using the above method.

METHOD 2: Measuring Volume with a Drop Counter

1. Obtain and wear goggles.
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 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.*
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. **CAUTION:** *Sodium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.*
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 Computers* folder. Check to see that the pH value is between 1.5 and 2.5.
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 . 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 . 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.
 - 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 . 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 . 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.



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* this jump.
2. Find and record the NaOH volume *after* the drop producing the largest pH increase was added.

Experiment 24

- Determine the volume of NaOH added at the equivalence point. To do this, add the two NaOH values determined above and divide by two.
- Calculate the number of moles of NaOH used.
- Using the equation for the neutralization reaction given in the introduction, determine the number of moles of HCl used.
- Calculate the HCl concentration using the volume of unknown HCl you pipeted out for each titration.
- If you did two titrations, determine the average [HCl] in mol/L.

DATA TABLE FOR PART 1


Mass of KHP (Formula Weight = 204.23)	g	g
NaOH volume added <i>before</i> the largest pH increase	mL	mL
NaOH volume added <i>after</i> the largest pH increase	mL	mL
Volume of NaOH added at equivalence point	mL	mL
Moles KHP	mol	mol
Moles NaOH	mol	mol
Concentration of NaOH	mol/L	mol/L
Average [NaOH]		M


DATA TABLE FOR PART 2


Molarity of NaOH (from Part 1, last line)	M	M
NaOH volume added <i>before</i> the largest pH increase	mL	mL
NaOH volume added <i>after</i> the largest pH increase	mL	mL
Volume of NaOH added at equivalence point	mL	mL
Moles NaOH	mol	mol
Moles acetic acid in unknown	mol	mol
Concentration of acetic acid in unknown	mol/L	mol/L
Average [acetic acid]		M

EQUIVALENCE POINT DETERMINATION: An Alternate 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 ($\Delta\text{pH}/\Delta\text{vol}$) 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 ($\text{pH}/\Delta\text{vol}$) on Page 2 of the experiment file by clicking on 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 ($\Delta^2\text{pH}/\Delta\text{vol}^2$) by clicking on the vertical-axis label, and choosing Second Derivative. In Method 2, view the second-derivative on Page 3 by clicking on the Next Page button, .

Prestudy

Name _____ Section _____

1.(5 points) A 0.5984 gram sample of acid potassium phthalate (GFW = 204.23) requires 19.35 mL of a NaOH solution to completely neutralize it using phenolphthalein as an indicator. What is the molarity of the NaOH solution?

2.(5 points) A 10.00 mL sample of an acetic acid, HC₂H₃O₂ solution requires 21.87 mL of a 0.1987 M NaOH solution to completely neutralize it using a phenolphthalein indicator. Calculate the molarity of the acetic acid solution.

