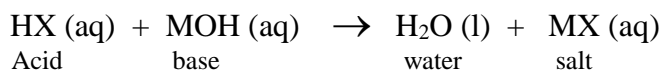


CHM 130LL: Vinegar Titration

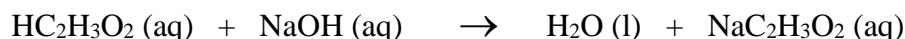
In one type of acid-base neutralization reaction, an acid can react with a metal hydroxide base to produce water and a salt:



The **protons** (H^+) from the acid react with the **hydroxide ions** (OH^-) from the base to form the water. The salt forms from the cation (usually a metal + ion) from the base and the anion (- ion) from the acid. Acids will react with bases whether the salt is soluble or insoluble, the salt's solubility does not determine whether the reaction occurs.

In this experiment, you will determine the molarity (or molar concentration) of acetic acid, $\text{HC}_2\text{H}_3\text{O}_2$ (aq), present in a sample of vinegar using a standard NaOH solution. (A **standard solution** has been analyzed, so its concentration is known to a certain degree of accuracy. In this experiment, the NaOH solution was standardized in our stockroom to four significant digits.) You will measure out a small volume of vinegar and use a **burette** to determine the volume of sodium hydroxide required to completely neutralize the vinegar. The process of slowly adding one solution to another until the reaction between the two is complete is called a **titration**.

The reaction between acetic acid, $\text{HC}_2\text{H}_3\text{O}_2$ (aq), and sodium hydroxide, NaOH (aq), is shown below:



When carrying out an acid-base neutralization reaction in the laboratory, you observe that most acid solutions and base solutions are colorless, and the resulting water and soluble salt solutions are also colorless. Thus, it is impossible to determine when a reaction has occurred, let alone when it is complete.

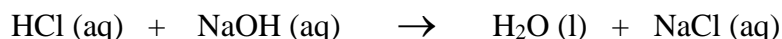
To monitor the progress of a neutralization reaction, you can use an **acid-base indicator**, a solution that changes color depending on the pH (or acid-content) of the solution. One commonly used indicator is *phenolphthalein*, which is colorless in acidic and neutral solutions and pink in basic (or alkaline) solution. During a titration, the indicator is added to the sample being analyzed. The **titrant** is slowly added to the sample until the **endpoint**, when the indicator just changes color, signaling that the reaction between the two is complete. Note that phenolphthalein however turns pink only when **excess** sodium hydroxide, NaOH (aq), has been added.

If the appropriate indicator has been chosen, the endpoint of the titration (i.e. the color change) will occur close to when the reaction is complete:

when moles of HCl = moles of NaOH in the example below

when moles of $\text{HC}_2\text{H}_3\text{O}_2$ = moles of NaOH in your titration

Titrating an Acid Consider the following reaction between hydrochloric acid, HCl (aq), and sodium hydroxide, NaOH (aq):



Using a standardized sodium hydroxide solution with a concentration of 1.020 M, a student titrated 25.00 mL of hydrochloric acid. (Molarity is moles per liter. In this example the NaOH has a molarity of **1.020 moles NaOH / 1 L NaOH = 1.020 M NaOH**). If 27.14 mL of sodium hydroxide was required to completely neutralize the hydrochloric acid to a phenolphthalein endpoint, calculate the molarity of the hydrochloric acid.

To determine the number of moles of hydrochloric acid, convert the volume of sodium hydroxide used to liters then multiply that with the molarity of sodium hydroxide (given as 1.020 M and shown below as a unit factor), as shown below. By showing the molarity explicitly as a fraction, you can see that the volume units (liters of NaOH) cancel. Since you actually need moles of hydrochloric acid, not moles of sodium hydroxide, you need to include one more step, the mole-to-mole ratio between sodium hydroxide and hydrochloric acid in the balanced equation which is 1 to 1.

$$27.14 \text{ mL NaOH} \left(\frac{1 \text{ L}}{1000 \text{ mL}} \right) \left(\frac{1.020 \text{ mol NaOH}}{1 \text{ L NaOH}} \right) \left(\frac{1 \text{ mol HCl}}{1 \text{ mol NaOH}} \right) = \mathbf{0.02768 \text{ mol HCl}}$$

Now take your answer for moles of HCl and divide by the volume of HCl in liters to get molarity.

$$\text{molarity of HCl} = \frac{\mathbf{0.02768 \text{ mol HCl}}}{\mathbf{0.02500 \text{ L HCl}}} = \mathbf{1.107 \text{ M HCl}}$$

Note that there are **4 significant figures** in all of the calculations for this experiment.

To find the molarity of the acetic acid in today's lab, you will need to follow the format of this calculation above using YOUR volumes of NaOH, the lab's molarity of NaOH written on the bottle, and the volume of acetic acid YOU used today. (Replace HCl with today's acid HC₂H₃O₂ in the calculations.)

The basic metric unit of volume is the liter (L), but the milliliter (mL) is most commonly used in lab. One L is equal to 1000 mL. Several types of apparatus are used to measure and deliver specific volumes.

Burettes are used when it is necessary to deliver a liquid to another container and record the amount delivered. A burette is marked in milliliters much like a graduated cylinder, except burette markings indicate the number of milliliters **delivered**. This means that **0** (none delivered) is at the top, and the numbers get larger as you go down the burette. The stopcock controls the liquid flow. It is open when parallel to the length of the burette and closed when perpendicular to the length of the burette.

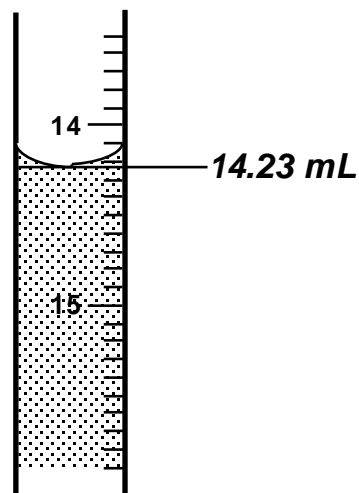
- **Condition the burette:** Obtain some deionized water in a small beaker. With the burette over the sink and the stopcock open, pour the water through the burette, letting it drain out the tip. After the burette is well-rinsed with DI water, close the stopcock and use the NaOH beaker to pour through the funnel 5-6 mL of the NaOH solution to be used into the burette. Tip the

burette sideways and rotate to completely rinse the inside of the burette. Drain this solution through the burette tip into the waste beaker. The last few mLs may not come out. Just turn upside down to empty.

- **Filling the burette:** Close the stopcock. Use the NaOH beaker to **slowly** fill the burette through the funnel to the zero mark. Place a waste beaker under the burette tip and open the stopcock slowly. The glass burette tip should fill with solution, leaving no air bubbles. If the tip does not fill with solution, ask the instructor for help. Continue to let out solution until the liquid level is around 3.00 mL. **But you record the exact volume to 2 decimal places!**
- **Reading the burette:** Record the volume by noting the bottom of the meniscus. (Be sure that the meniscus is at eye level). Read the burette to 2 decimal places, such as 0.12 or 0.47 mL. NOTE – your burette volumes should NOT all end in zero.

Thus, in the example at the right, the meniscus is about one-third of the way between 14.2 and 14.3, so the volume is recorded as **14.23 mL**.

Burette readings are always recorded in mL to two decimal places.



Analysis of Vinegar

CAUTION: SODIUM HYDROXIDE, NaOH, CAN CAUSE CHEMICAL BURNS AND DAMAGE EYES VERY QUICKLY. ANY NaOH SPILLED ON YOUR SKIN MUST BE RINSED IMMEDIATELY WITH WATER FOR 15 MINUTES. ANY NaOH SPILLED ON THE LAB BENCHES SHOULD BE NEUTRALIZED, THE AREA RINSED WITH WATER AND WIPED CLEAN. INFORM YOUR INSTRUCTOR OF ANY NaOH SPILLS.

WEAR GOGGLES AT ALL TIMES, even when you are washing the glassware to avoid exposing your eyes to NaOH solution. Wash your hands completely with soap and water before leaving the lab today.

Today you will be working alone without a partner. Read directions carefully.

1. Make sure your 150-mL beaker is clean and dry. Obtain about **50 mL** of vinegar in your 150-mL beaker using the markings on the beaker. Obtain a 10.00-mL pipet and pipet pump. Your instructor will demonstrate how to use the pipet pump to draw the vinegar solution up the pipet so that the bottom of the meniscus is exactly at the 10.00 mL mark on the pipet. Condition the 10.00-mL pipet with a small amount of vinegar solution (ie rinse the pipet with a small amount of DI water then a small amount of vinegar). Your instructor will show you how.
2. Label your three 125-mL Erlenmeyer flasks 1, 2, and 3. Use the pipet pump to pipet 10.00 mL of vinegar into each of the three Erlenmeyer flasks. Add ~25 mL of deionized water to each flask using a graduated cylinder. The water does not affect the amount of vinegar present, so it does not have to be exactly 25 mL. (The water is added so the stir bar won't splash when stirring.)

3. Obtain a burette and clamp it to the ring stand using a burette clamp, an X-shaped clamp that can hold two burettes.
4. Next, obtain about **100 mL** of the NaOH solution in your 250 mL clean, dry beaker using the markings on the beaker. **Record the molarity of the NaOH solution from the bottle on your Report Sheet.** Condition your burette as on page 2-3. This prevents dilution of your solution by any water droplets that remain inside the burette. Use your 400-mL beaker as a waste container.
5. Close the stopcock, and attach the burette onto the stand using the burette clamp. Place the 400-mL beaker under the burette, and fill your burette with your NaOH solution as per page 3.
6. **Read the initial volume of NaOH** as per page 3 and record directly onto your Data Sheet.
7. Add 2-3 drops of phenolphthalein indicator to each of your vinegar solutions in the flasks. Obtain a stir plate and a stir bar (the small white magnet). Set up your stir plate under your burette, and turn the dial to OFF. Holding flask #1 at an angle, slowly lower the stir bar into the flask, so none of the vinegar solution splashes out. Place the flask onto the middle of the stir plate. Adjust the dial on the stir plate until the bar stirs the solution briskly but smoothly.
8. Position flask #1 under the burette. Open the stopcock, so a continuous stream of NaOH flows into the flask. Allow about 8 mL of NaOH to flow quickly into your flask, and then allow the solution to flow slower **until you begin seeing flashes of pink. At this point adjust the stopcock, so the NaOH is delivered much more slowly.** When the pink color persists for a few seconds, adjust the stopcock until the NaOH is delivered **drop by drop**. Keep your hand on the stopcock, and prepare to turn it off when the solution turns permanently pink. Often one drop will be enough. Let the solution sit for 30 seconds to insure that the pink color does not go away. If it does, add one more drop and wait again for 30 seconds.
9. **Record the final volume of NaOH** (as per page 3) on your Data Sheet. To get the volume of solution delivered, subtract the initial volume from the final volume.
10. Use the tweezers/forceps to retrieve the stir bar from flask #1. Thoroughly rinse the stir bar, then slowly lower it into flask #2. Pour the solution from flask #1 into the waste beaker. Refill the burette with NaOH to near 3.00 mL (you do not want to run out during a trial – refilling in the middle of a trial reduces accuracy), and record the initial volume for trial #2 on your Data Sheet. Repeat the titration for trial #2. Record your final volume of NaOH on your Data Sheet.

Note: Let your instructor know if the stir bar accidentally falls out and goes down the drain when you are rinsing your flask. We have a magnetic stick that will allow us to retrieve the magnetic stir bar.

11. Dispose of the solution in flask #2 into the waste beaker, and retrieve the stir bar. Thoroughly rinse the stir bar, and slowly lower it into flask #3. Refill the burette to around 3.00 mL, and record the initial volume for trial #3 on your Data Sheet. Repeat the titration for trial #3. Record your final volume of NaOH on your Data Sheet.
12. Dispose of the solution in flask #3 into the waste beaker, and retrieve the stir bar. If all three of your trials resulted in a slightly pink endpoint, you are done. If not and there is time, do a fourth/fifth/sixth trial. You are graded on accuracy this lab and so a poor trial may affect your grade.

13. When you are finished with three good trials which are pale pink not hot pink, empty the burette, and rinse it with tap water, allowing some tap water to run through the tip then return it. Rinse the pipette with tap water and return it.
14. Combine any remaining vinegar and NaOH solutions in your large waste beaker, so they will neutralize each other. Dispose of them in the hood waste container. Thoroughly rinse your flasks, and return them to their proper places. Wash and dry the stir bar and return it to its proper place.

BE SURE TO WASH AND DRY YOUR LAB BENCH AFTER COMPLETING THE EXPERIMENT TO REMOVE ALL TRACES OF ANY SPILLED CHEMICALS.

WASH YOUR HANDS COMPLETELY WITH SOAP AND WATER BEFORE LEAVING THE LAB.

You must wear your goggles until everyone in lab is finished. Calculate the molarity of the vinegar solution as shown in the Example Calculation on page 2 highlighted in yellow. Be sure to express all measurements with the correct units and to the correct number of significant figures. Calculate the average molarity for all three of your trials. Finish your report sheet and turn it in to your instructor.

CHM 130LL: Vinegar Titration

Name: _____

You work ALONE this week!

Section Number: _____

Analysis of Vinegar

Molarity of NaOH (from bottle), M _____

| | Trial 1 | Trial 2 | Trial 3 |
|---|----------|----------|----------|
| Volume of vinegar, mL | 10.00 mL | 10.00 mL | 10.00 mL |
| Volume of vinegar, L (watch sig fig) | | | |
| Final burette reading, mL | | | |
| Initial burette reading, mL | | | |
| Volume of NaOH used, mL (subtract initial from final) | | | |
| Molarity of $\text{HC}_2\text{H}_3\text{O}_2$, M | | | |
| Average Molarity of $\text{HC}_2\text{H}_3\text{O}_2$, M | | | |

Show your Molarity calculations for all three trials below.

(trial 1)

(trial 2)

(trial 3)

Questions:

1. Fill in the blanks for the following by circling the answer for each missing word:

- a. Phenolphthalein is _____ in acidic solutions, colorless pink
 _____ in neutral solutions, colorless pink
 and _____ in basic solutions. colorless pink
- b. Acidic solutions contain _____ ions, and H^+ OH^-
 basic solutions contain _____ ions. H^+ OH^-

2. Explain why the solution being titrated first turns pink then goes colorless before the endpoint is reached.

3. Before the endpoint is reached is the solution acidic or basic? _____

4. Before the endpoint is reached does the solution contain more acid or base? _____

5. Explain why the solution being titrated turns pink and stays pink at the endpoint.

6. At the endpoint is the solution acidic or basic? _____

7. At the endpoint does the solution contain more acid or base? _____

8. A student gets a dark pink color for trial 2 but uses that data to calculate the molarity of the acetic acid. Is their molarity value too high or too low? Explain and be specific