Experiment 16

Titration of Vinegar

Pre-Lab Assignment

Before coming to lab:

- Read the lab thoroughly.
- Answer the pre-lab questions that appear at the end of this lab exercise.

Purpose

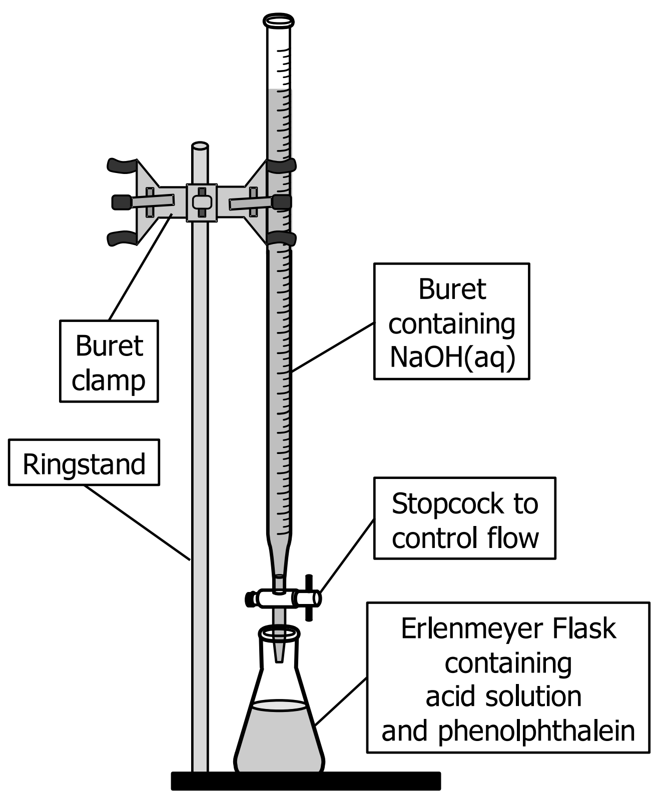
- Gain proficiency in performing titrations
- Determine the molar concentration and mass/mass percent concentration of acetic acid in an unknown vinegar solution

Background

In this experiment we will perform a **titration**, a lab technique that is used to determine the concentration of a solute in a solution. We will use titration to find the molar concentration and mass/mass percent of acetic acid in vinegar.

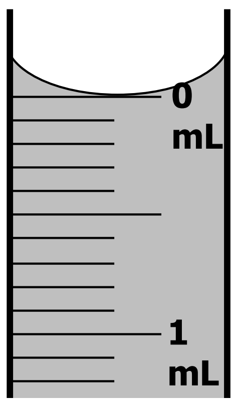
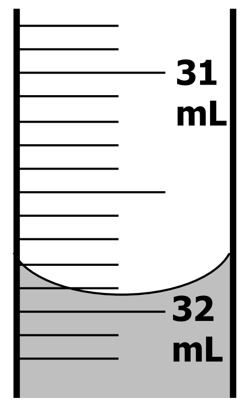
During the titrations in this experiment we will neutralize an acid solution by slowly adding NaOH solution using a buret. A **buret** is a tool used to dispense a precise amount of solution. An indicator is also added to the acid solution to signal the end the titration (the **endpoint**). An **indicator** is a dye that changes color at the endpoint of a titration. At the endpoint for an acid-base titration, all of the acid has been neutralized and no more NaOH solution is added.

The indicator that we will use in this experiment is phenolpthalein. Phenolpthalein is colorless in acidic solutions and pink in basic solutions. At the beginning of the titration the acid solution with phenolphthalein is clear and colorless. When the titration is near the endpoint, flashes of pink will appear in the acid solution. At the endpoint of the titration only one drop of NaOH solution is needed to turn the acid solution a permanent pink. The amount of NaOH solution used to neutralize the acid can be determined using readings from buret at the beginning and end of the titration.



Reading a Buret

Burets are designed to deliver precise volumes of liquids. Each marking on the buret represents 0.1 mL, so volume readings must be estimated to the nearest 0.01 mL (all reported volumes must have two decimal places). Note that the zero mark is at the top of the buret instead of at the bottom and that the numbers increase as you move down the buret (this is the opposite of a graduated cylinder). Make sure your eye is level with the meniscus and take the reading from the bottom of the meniscus. Examples are shown below.

Initial Buret Reading	Final Buret Reading
 **CORRECT:** 0.00 mL ☺ **INCORRECT:** 0 mL ☹ 0.0 mL ☹	 **CORRECT:** 31.93 mL ☺ **INCORRECT:** 32.07 mL ☹

The total volume of liquid dispensed from the buret is calculated as:

final buret reading – initial buret reading = 31.93 mL – 0.00 mL =31.93 mL

In Part A we will prepare and standardize a solution of NaOH. **Standardization** is process of determining the exact concentration of a solution by titration. The exact concentration of the NaOH solution will be determined by titrating a solution containing a known amount of potassium hydrogen phthalate (abbreviated KHP) dissolved in water. The balanced equation for the reaction between KHP and NaOH is shown below:

$$\underset{\text{(KHP)}}{KHC_8H_4O_4(aq)} \quad + \quad NaOH(aq) \quad \rightarrow \quad KNaC_8H_4O_4(aq) \quad + \quad H_2O(\ell)$$

By knowing the volume of NaOH solution dispensed from the buret and the mass of KHP used, we can calculate the molarity of the NaOH solution. A sample calculation to determine the molarity of NaOH is shown in Example 1.

Example Problem 1: Molarity of Standardized NaOH

A 0.9341 g sample of KHP (204.23 g/mol) is dissolved in water and titrated with 20.61 mL of NaOH solution. Calculate the molarity of the NaOH solution.

$$\underset{\text{(KHP)}}{KHC_8H_4O_4(aq)} \quad + \quad NaOH(aq) \quad \rightarrow \quad KNaC_8H_4O_4(aq) \quad + \quad H_2O(\ell)$$

Step 1: Convert grams of KHP to moles of NaOH using the balanced equation for the reaction of KHP with NaOH.

$$0.9341\ \text{g}\ \left(\frac{1\ \text{mol KHP}}{204.23\ \text{g KHP}}\right)\left(\frac{1\ \text{mol NaOH}}{1\ \text{mol KHP}}\right) = 0.004574\ \text{mol NaOH}$$

Step 2: Convert mL of NaOH solution given in the problem to L of NaOH. Use the moles of NaOH from Step 1 and L of NaOH to calculate the molarity of NaOH (mol/L).

$$20.61\ \text{mL NaOH solution}\ \left(\frac{1\ \text{L}}{1000\ \text{mL}}\right) = 0.02061\ \text{L NaOH solution}$$

$$\frac{0.004574\ \text{mol NaOH}}{0.02061\ \text{L NaOH solution}} = 0.2219\ \frac{\text{mol NaOH}}{\text{L solution}} = \ 0.2219\ \text{M NaOH}$$

In this example, the concentration of NaOH is 0.2219 M.

In Part B we will use the standardized NaOH from Part A to determine the concentration of acetic acid in a vinegar solution. The balanced equation for the neutralization reaction between acetic acid and NaOH is shown below.

$$\underset{\text{(acetic acid)}}{HC_2H_3O_2(aq)} \quad + \quad NaOH(aq) \quad \rightarrow \quad NaC_2H_3O_2(aq) \quad + \quad H_2O(\ell)$$

By knowing the volume and molarity of the NaOH dispensed from the buret and the volume of vinegar solution, we can determine the molarity of acetic acid in vinegar. The molarity of acetic acid can be converted to the mass/mass percent concentration using the density of the vinegar (1.01 g/mL) and the molar mass of acetic acid (60.06 g/mol). A sample calculation for calculating the molarity and mass/mass percent of acetic acid in vinegar is shown in Example 2.

Example Problem 2: Molarity and mass/mass percent of acetic acid in vinegar

The titration of 10.00 mL of vinegar requires 31.93 mL of 0.2219 M NaOH to reach the endpoint. Calculate the molarity and mass/mass percent of acetic acid in the vinegar.

$$HC_2H_3O_2(aq) \quad + \quad NaOH(aq) \quad \rightarrow \quad NaC_2H_3O_2(aq) \quad + \quad H_2O(\ell)$$

(acetic acid)

Step 1: Convert the volume of NaOH solution to moles of $HC_2H_3O_2$ using the molarity of NaOH and the balanced equation for the reaction of $HC_2H_3O_2$and NaOH.

$$31.91\text{ mL NaOH}\left(\frac{1\text{ L NaOH}}{1000\text{ mL NaOH}}\right)\left(\frac{0.2219\text{ mol NaOH}}{1\text{ L NaOH}}\right)\left(\frac{1\text{ mol HC}_2\text{H}_3\text{O}_2}{1\text{ mol NaOH}}\right)$$

$$= 0.007085\text{ mol HC}_2\text{H}_3\text{O}_2$$

Step 2: Use the moles of $HC_2H_3O_2$ from Step 1 and L of vinegar to calculate the molarity of $HC_2H_3O_2$ (mol/L).

$$10.00\text{ mL vinegar solution}\left(\frac{1\text{ L}}{1000\text{ mL}}\right) = 0.01000\text{ L vinegar solution}$$

$$\frac{0.007085\text{ mol HC}_2\text{H}_3\text{O}_2}{0.01000\text{ L vinegar solution}} = 0.7081\ \frac{\text{mol HC}_2\text{H}_3\text{O}_2}{\text{L solution}} = \ 0.7085\text{ M HC}_2\text{H}_3\text{O}_2$$

Step 3: Use the density of the vinegar (1.01 g/mL) and the molar mass of acetic acid (60.06 g/mol) to convert the molarity of $HC_2H_3O_2$ to mass/mass %.

$$\frac{0.7085\text{ mol HC}_2\text{H}_3\text{O}_2}{1\text{L vinegar solution}}\left(\frac{1\text{ L}}{1000\text{ mL}}\right)\left(\frac{60.06\text{ g HC}_2\text{H}_3\text{O}_2}{1\text{ mol HC}_2\text{H}_3\text{O}_2}\right)\left(\frac{1\text{ mL vinegar solution}}{1.01\text{ g vinegar solution}}\right)\times 100\%$$

$$= 4.21\text{ m/m\% HC}_2\text{H}_3\text{O}_2$$

In this example, the concentration of $HC_2H_3O_2$ in vinegar is 0.2219 M or 4.21 m/m %.

Procedure

Part A: Standardization of NaOH Solution

Safety: Wear safety googles at all times.

If you come into contact with 6.0 M NaOH, wash the area effected immediately with water.

1. Obtain the following equipment from the side counters or the instructor's desk:
   - 50-mL buret
   - 10-mL volumetric pipette
   - pipette bulb
   - ringstand with buret clamp

2. Take out a 250-mL beaker from your drawer and use a pencil to label it as your temporary waste container on the large white marking spot.

Preparing the KHP solutions

3. Label two 125-mL Erlenmeyer flasks as #1 and #2 on the white marking spot. Add about 1 g of solid KHP to each flask, making sure to record the exact masses of KHP used on your report sheet. Add about 25 mL of DI water using a graduated cylinder and 2-3 drops of phenolphthalein indicator to each flask and swirl to dissolve the KHP. If the KHP does not dissolve, it may be necessary to heat the flask slightly on a hot plate to insure the KHP dissolves.

Preparing the NaOH solution

4. Fill a 500 mL Florence flask about halfway with DI water (about 250 mL). Use a graduated cylinder to measure out and add 20 mL of 6 M NaOH to the Florence flask. Seal the flask with a rubber stopper and gently swirl to mix the solution.

Preparing the buret

5. Close the stopcock on the buret and use a funnel to pour about 10 mL of NaOH solution into the buret. Remove the funnel and gently tilt the buret on its side and roll the liquid around to rinse the entire inner surface. Allow some of the NaOH solution to drain out through the tip into the waste beaker at your station. Flip over the buret and pour the remaining NaOH rinse into your waste beaker. *Repeat this step a second time.*

6. Place the empty buret in the buret clamp on your ringstand. Put the ringstand in the nearest sink so that the top of the buret is near eye level. Check that the stopcock on the buret is closed. Fill the buret with NaOH solution to just above the 0.00-mL mark. Use a funnel and pour very slowly!

7. Place the Florence flask containing the NaOH under the buret. Open the stopcock slowly and let the liquid level drop so that the bottom of the meniscus rests on the 0.00 mL mark. Make sure there are no air bubbles in the tip of the buret. Remove the buret and ringstand from the sink

   If you are having trouble removing air bubbles from the buret tip or the NaOH does not flow out of the buret in a steady stream, ask your instructor for help.

8. Since the bottom of the meniscus of the NaOH in the buret is resting on the 0.00-mL mark, record 0.00 mL as the **initial buret reading**.

Performing the titration

9. Place the acid solution in the Erlenmeyer flask under the buret filled with base. Begin the titration by slowly adding 1 mL base from the buret to the acid solution in the Erlenmeyer flask. Swirl the Erlenmeyer flask after you add the base so the chemicals are well-mixed. Continue to add 1 mL portions of base to the flask, swirling after each addition. A small amount of pink may briefly appear briefly in the flask when the base is added, but it will quickly disappear as the flask is swirled.

10. As you approach the end of the titration, the pink color in the flask will take longer to disappear. When this happens, begin adding the NaOH solution **drop by drop**, swirling the flask after adding each drop. Stop when the addition of just one drop of NaOH solution turns the solution in the Erlenmeyer flask a pale pink color that does not disappear when the flask is swirled. This indicates that you have reached the endpoint of the titration.

11. Use the bottom of the meniscus of the NaOH solution in the buret determine the **final buret reading** to two places past the decimal and record the value on your report sheet.

12. Refill your buret with NaOH solution to 0.00-mL mark and repeat the titration for the second sample of KHP.

13. When you are finished with both titrations, dispose of your neutral pink solutions in the sink with plenty of water. Rinse out the flasks with DI water to reuse for part B.

14. Calculate the molarity of the NaOH solution for each trial and the average molarity.

Part B: Titration of Vinegar

15. Obtain about 50 mL of vinegar in a clean, dry 100-mL beaker.

16. Rinse the inside of the 10-mL volumetric pipette with vinegar. Use the pipette bulb to draw the vinegar into the pipette until the bulb is half-filled. Quickly remove the pipette bulb and place your finger on the top of the pipette to hold in the liquid. Gently the tilt the pipette to one side and roll the liquid around to rinse the entire inner surface. Let the vinegar rinse drain out of the bottom of the pipette into your temporary waste beaker. *Repeat this step one more time.*

17. Use the volumetric pipette to transfer 10.00-mL of vinegar to both of your labeled 125-mL Erlenmeyer flasks. Your instructor will show you how to use the pipette. Don't forget to make sure that the bottom of the meniscus is resting on the calibration mark and there are no air bubbles in the pipette before transferring the vinegar to the flask.

    Note: You can reuse your empty Erlenmeyer flasks from the titrations of KHP. You do not need to dry the flasks before using them.

18. Record 10.00 mL as the volume of vinegar on your report sheet for both samples. Then add about 20 mL of DI water and 2 to 3 drops of phenolphthalein to the Erlenmeyer flask.

19. Titrate the vinegar samples following steps 9-13 of this procedure (the same as you did for the KHP samples). Discard the pink solutions in the sink with plenty of water.

20. Rinse the buret and volumetric pipette with DI water and return all titration equipment to the side counters and instructor's desk.

21. Empty your temporary waste beaker in the hazardous waste jug in the fumehood. Wash your dirty glassware and return it to your drawer.

22. Calculate the molarity and mass/mass percent of acetic acid in your vinegar sample.

Experiment 16—Report Sheet Name: ______________

Part A: Standardization of NaOH Solution

	Trial 1	Trial 2	Trial 3
1. Mass of Erlenmeyer flask + KHP (g)	________	________	________
2. Mass of Erlenmeyer flask (g)	________	________	________
3. Mass of KHP (g)	________	________	________
4. Final buret reading (mL)	________	________	________
5. Initial buret reading (mL)	________	________	________
6. Volume of NaOH used (mL)	________	________	________

Show the calculation of the molarity of NaOH for Trial 1 (see Example 1).

	________	________	________
7. Molarity of NaOH (mol/L)	________	________	________
8. Average molarity of NaOH (mol/L)	________		

Part B: Titration of Vinegar

Average molarity of NaOH solution from Part A (mol/L) ________________

	Trial 1	Trial 2	Trial 3
1. Volume of vinegar solution (mL)	________________	________________	________________
2. Final buret reading (mL)	________________	________________	________________
3. Initial buret reading (mL)	________________	________________	________________
4. Volume of NaOH used (mL)	________________	________________	________________

Show the calculation for the molarity of acetic acid for Trial 1. (See Example 2.)

	________________	________________	________________
5. Molarity of $HC_2H_3O_2$ (mol/L)	________________	________________	________________
6. Average molarity of $HC_2H_3O_2$ (mol/L)	________________		

Show the calculation for the mass/mass percent of acetic acid for Trial 1. Assume the density of vinegar is 1.01 g/mL. (See Example 2.)

7. Mass/mass percent $HC_2H_3O_2$ (%)	________________	________________	________________

8. Average mass/mass percent $HC_2H_3O_2$ (%)

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Experiment 16—Post-Lab Assignment. **Name: ______________________________**

1. Consider the possible sources of experimental error listed below. Circle the letter(s) of the errors that will NOT affect the final results of this experiment.

   a. The sodium hydroxide is not completely mixed with the water in the Florence flask.

   b. The buret is wet on the inside with water and is not rinsed with NaOH solution before it is filled with NaOH and the titration is started.

   c. The KHP samples are dissolved in 50 mL of water instead of 25 mL of water.

   d. 5 drops of phenolphthalein are used instead of 2-3 drops.

   e. An air bubble that was trapped in the tip of the buret comes out during the titration.

   f. The Erlenmeyer flasks are not dried before the vinegar samples are prepared for titration.

2. A 10.0-mL sample of household ammonia solution required 18.15 mL of 0.320 M HCl to be neutralized.

$$HCl(aq) + NH_3(aq) \rightarrow NH_4Cl(aq)$$

   a. Calculate the molarity of the ammonia.

   b. The density of the ammonia solution is 0.990 g/mL. The molar mass of ammonia is 17.04 g/mol. Calculate the mass/mass percent of ammonia in the solution.

3. A hydrochloric acid solution is standardized using 0.500 g of sodium Na_2CO_3. Find the molarity of the acid if 29.50 mL are required to reach the endpoint.

$$2\ HCl(aq) + Na_2CO_3(s) \rightarrow 2\ NaCl(aq) + H_2O(\ell) + CO_2(g)$$

4. If 22.15 mL of 0.100 M sulfuric acid is required to neutralize 10.0 mL of lithium hydroxide solution, what is the molar concentration of the base?

$$H_2SO_4\ (aq) + 2\ LiOH(aq) \rightarrow Li2SO4(aq) + H_2O(\ell)$$

5. A Rolaids tablet contains calcium carbonate, which neutralizes stomach acid. IF a Rolaids tablet neutralizes 42.15 mL of 0.320 M hydrochloric acid, who many milligrams of calcium carbonate are in a Rolaids tablet?

$$CaCO_3(s) + 2\ HCl(aq) \rightarrow CaCl_2(aq) + H_2O(\ell) + CO_2(g)=$$

Name: ________________________

Pre-lab Assignment for Titration of Vinegar

1. In your own words, define the following chemistry terms.

a. Endpoint

b. Indicator

c. Molar concentration (M)

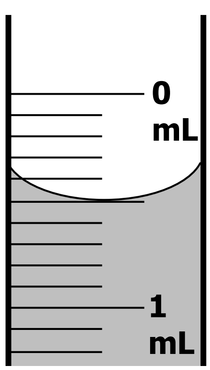
d. Mass/mass percent concentration (m/m %)

e. Standardization

f. Titration

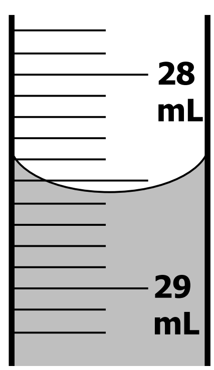
2. Read the burets to the correct number of decimal places. Examples are given in the Background section of this experiment.

a.



Volume ____________________

b.



Volume ____________________

3. When approaching the endpoint for the titration, how can you tell that you are very close?

4. A 1.015-g sample of KHP (204.23 g/mol) is dissolved in water and titrated with 24.55 mL of NaOH solution. Calculate the molarity of the NaOH solution. (See Example 1.)

5. The titration of 10.00 mL of vinegar requires 41.60 mL of 0.202 M NaOH to reach the endpoint.

a. Calculate the molarity of acetic acid in the vinegar. (See Example 2.)

b. Assume the density of the vinegar solution is 1.01 g/mL. The molar mass of acetic acid ($HC_2H_3O_2$) is 60.06 g/mol. Calculate the mass/mass percent of acetic acid in the vinegar. (See Example 2.)