

Experiment 12

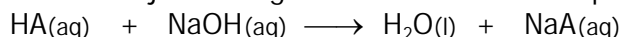
Chem 110 Lab

TITRATION

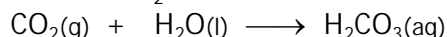
I. INTRODUCTION

In this experiment you will be determining the molarity of an unknown acid solution by measuring the volume of a sodium hydroxide solution of known concentration required to neutralize a measured volume of the unknown acid solution.

Your unknown acid is a monoprotic (only one acidic hydrogen) acid. Let's give it the formula HA. You will accurately measure about 20 mL of the unknown acid solution, add one drop of indicator (see definition below) and then add just enough NaOH solution to completely neutralize the acid:



The indicator is added because there is no observable change that occurs when the neutralization reaction is complete. The indicator used in this experiment, phenolphthalein, is colorless in acidic solution but turns pink when there is an excess of base present, therefore you will know that the reaction is complete when you see the first faint hint of pink color in the reaction mixture. Because the pink color occurs when there is an excess of base (more than is needed to completely react all of the acid) present, the lighter the pink color is at the end point, the better. Also, the pink should last for 30 seconds. After that time it may fade due to the presence of carbonic acid, H_2CO_3 , in the solution. Carbonic acid is formed when CO_2 from the air dissolves in and then reacts with water:



The H_2CO_3 reacts to neutralize the NaOH that was causing the pink color, and the pink color goes away.



The technique used to measure the volume of sodium hydroxide solution required to react with the acid solution is called titration.

TITRATION is the process of the gradual addition of a standard solution (in this case a solution of NaOH) to a second solution until all of the solute in the second solution has completely reacted.

STANDARD SOLUTION is a solution of known concentration. The standard solution in this experiment is the NaOH solution.

TITRANT is the name given to the solution that is gradually added to the second solution. The titrant in this experiment is the NaOH solution.

BURET is an instrument used to measure volume; a graduated glass cylinder with a stopcock on one end. The volume measurement is made by reading the fluid level in the buret before and after the fluid in the buret is dispensed through the stopcock.

INDICATOR is a substance which is added to the reaction system in small amounts; it indicates that the reaction is complete (has reached the end point of the reaction) by changing color. The indicator used in this experiment is phenolphthalein.

END POINT is the stage in the titration at which the indicator color change is observed, indicating that the reaction is complete.

KNOW THESE DEFINITIONS WELL FOR THE EXPERIMENT QUIZ

II. EXPERIMENT

CAUTION

- The solution of sodium hydroxide, NaOH, can harm your skin and your eyes. Any base solution spilled on your skin or splashed into your eyes should be rinsed immediately with a large volume of water.
- Solutions of acids can harm your eyes, skin, and clothing. Handle with care. Any acid solution spilled on your skin or splashed into your eyes should be rinsed immediately with a large volume of water.

Lab 1

- View film: "Use of a Buret"
- Practice reading the buret.

A. Preparation of Unknown Acid and Standard Base Solutions

1. Clean and dry two 125 mL Erlenmeyer flasks. With pencil, write an A (for acid) on the white patch on one of the flasks. On the white patch on the other flask write B.
2. Take flask A and this paper to your instructor and get your unknown acid solution. Write your unknown number below. Immediately stopper flask A.
Unknown # _____
3. Take flask B and this paper to the reagent bench and get about 40 mL of the standard NaOH solution. Write its concentration below. Immediately stopper flask B.

Molarity of Standard NaOH Solution _____ M

Lab 2

B. Preparation of Burets

1. Get a buret clamp from the community drawer at your bench. Attach the buret clamp to a ring stand.
2. From the stockroom check out a 250 mL Erlenmeyer flask and two (2) 25 mL burets. Attach the burets to the buret clamp.
3. Rinse both burets (one at a time) as follows:

Do the following at your bench, using your largest beaker as a waste container. (Be sure to label the beaker "waste".) With its stopcock closed, add approximately 10 mL of deionized water to the buret, and tilt and rotate the buret in an almost horizontal position to rinse the entire inside wall. Pour about half the water out the top of the buret and then open the stopcock and allow the rest of the water to drain through the buret tip into the waste beaker. Repeat this rinsing process two (2) more times.

4. Preparation of the unknown acid buret
 - a. To one of the burets, stopcock closed, add approximately 5 mL of your unknown acid solution. Tilt and rotate the buret to rinse the entire inside wall with the unknown acid solution.
 - b. Attach the buret to the buret clamp and place the card labeled "Acid" under the buret. (Find "Acid" and "Base" cards on the reagent bench.) Place the waste beaker under the buret, and open the stopcock to allow the acid rinse solution to drain through the stopcock. (Don't forget to close the stopcock again.)

- c. Add unknown acid solution to the buret until it is slightly above the zero mark on the buret. Open the stopcock to allow some solution to drain into the buret tip. Check to be sure that the tip is completely filled and that there are no air bubbles in it. At this point the solution in the buret should be at or below the zero mark.
5. Preparation of the standard base buret
 - a. To the other buret, stopcock closed, add approximately 5 mL of the standard NaOH solution. Tilt and rotate the buret to rinse the entire inside wall with the NaOH solution.
 - b. Attach the buret to the buret clamp and place card labeled "Base" under the buret. Place the waste beaker under the buret, and open the stopcock to allow the NaOH rinse solution to drain through the stopcock. (Don't forget to close the stopcock again.)
 - c. Add NaOH solution until it is slightly above the zero mark on the buret. Open the stopcock to allow some solution to drain into the buret tip. Check to be sure that the tip is completely filled and that there are no air bubbles in it. At this point the solution in the buret should be at or below the zero mark.

C. TITRATION

1. Use a buret reading card to help you read the "initial volume" of the solution in each buret. (Don't forget to read to two (2) decimal places!) Record these readings in table 12.1. on the Report Sheet. If you aren't sure that you are reading the buret correctly, ask your instructor to check the reading for you.

DON'T FORGET TO TAKE YOUR INITIAL BURET READINGS!!!!

2. Clean the 250 mL Erlenmeyer flask, giving it a final rinse with deionized water. Place the Erlenmeyer flask under the unknown acid buret, and lower the buret so that its tip is inside and about a half inch below the top of the flask. Open the stopcock and allow approximately 20 mL of your unknown acid to drain rapidly out of the buret into the Erlenmeyer flask.
3. Add 1 or 2 drops of phenolphthalein indicator (in a labeled dropper bottle at your bench) to the acid solution and swirl gently to mix.

DON'T FORGET TO ADD INDICATOR!!!!

4. Place the flask containing the acid solution under the NaOH buret. Titrate the unknown acid with the NaOH solution by allowing it to drain slowly out of the buret. Swirl the flask continuously as you add the NaOH solution. As the base is added you will observe a pink color localized at the spot the NaOH enters the solution that disappears rapidly. As you approach the end point, the pink color will take longer to disappear. When this occurs, slow the rate of NaOH addition to drop by drop. When you are near the end point, rinse the inside walls of the Erlenmeyer flask with deionized water to rinse any splashed acid solution off the walls. When you add the final drop (or even half-drop) and the pink color remains for at least 30 seconds, you have reached the end point of the titration.

NOTE: If you "over-shoot" the end point (add too much NaOH solution), add a little more of your unknown acid solution to the flask (until the solution becomes colorless again) and then add NaOH again until a proper end-point is reached.

Never allow the liquid in the buret to drop below the 25 mL mark.

5. Make the final readings for both burets and record them in table 12.1 on the Report Sheet.

DON'T FORGET TO TAKE YOUR FINAL BURET READINGS!!!!

6. Refill both burets, rinse the 250 mL Erlenmeyer flask with deionized water and carry out a second and then a third titration (Steps 1-5)

D. CALCULATIONS

1. For each titration, carry out the following calculations:
 - a. Calculate the volume of the NaOH solution and the volume of the unknown acid solution, giving the setup in Table 12.1, page 5.
 - b. Calculate the moles of NaOH used to neutralize the acid, writing the setup in Table 12.2, page 6.
 - c. Calculate the moles of acid, writing the setup in Table 12.2, page 6.
 - d. Calculate the molarity of the acid solution, writing the setup in Table 12.2, page 6.
2. Calculate the average molarity (you must have a minimum of 3 trials) and report it on page 7.
3. Get the "correct" molarity for your unknown acid from your instructor and calculate the percent relative error in your experimental (your average) molarity.

If your percent error is greater than 2% you must perform more titrations.

At the end of lab, return the burets and 250 mL Erlenmeyer flask to the stockroom. Before returning the burets to the stockroom, be sure to rinse them thoroughly with deionized water.

Report Experiment 12

Chem 110

TITRATION

Name _____ Date _____
(last) (first)

Instructor's Initials _____

A. DATA & CALCULATIONS

Unknown Acid# _____ Molar Concentration of Standard NaOH Solution: _____

Table 12.1

Titration # →	1		2		3	
	Acid	Base	Acid	Base	Acid	Base
Final Reading						
Initial Reading						
Calculated Volume (mL)						
Volume in Liters						

If necessary

Titration # →	4		5		6	
	Acid	Base	Acid	Base	Acid	Base
Final Reading						
Initial Reading						
Calculated Volume (mL)						
Volume in Liters						

Table 12.2 (Give setups of calculations in the table.)

TITRATION #1	
MOLES OF NaOH	
MOLES OF UNKNOWN ACID	
MOLARITY OF UNKNOWN ACID SOLUTION	
TITRATION #2	
MOLES OF NaOH	
MOLES OF UNKNOWN ACID	
MOLARITY OF UNKNOWN ACID SOLUTION	
TITRATION #3	
MOLES OF NaOH	
MOLES OF UNKNOWN ACID	
MOLARITY OF UNKNOWN ACID SOLUTION	
TITRATION #4, if necessary	
MOLES OF NaOH	
MOLES OF UNKNOWN ACID	
MOLARITY OF UNKNOWN ACID SOLUTION	
TITRATION #5, if necessary	
MOLES OF NaOH	
MOLES OF UNKNOWN ACID	
MOLARITY OF UNKNOWN ACID SOLUTION	
TITRATION #6, if necessary	
MOLES OF NaOH	
MOLES OF UNKNOWN ACID	
MOLARITY OF UNKNOWN ACID SOLUTION	

1. Average Molarity of Unknown Acid Solution

Unknown Acid # _____

2. Correct Molarity of Acid (From Instructor): _____

3. Percent Error:

B. Problems and Questions:

1. What is the molar concentration of an aqueous solution of oxalic acid if 25.00 ml of 0.2500 M NaOH is required to reach the end point in titrating 20.00 mL of the oxalic acid solution? (Don't forget to first write and balance the equation for the reaction.)

2. Consider each of the following and decide what effect it would have on your experimental molarity. Will the error cause your answer (the molarity of the unknown acid solution) to be higher or lower than it should be, or will it have no effect at all.

a. Adding your unknown acid to an Erlenmeyer flask that already has 2 mL of deionized water in it. a. _____

b. Using the wrong concentration, 0.9850 M NaOH, in your calculations instead of the correct concentration, 1.300 M NaOH. b. _____

c. Using 21.99 mL of unknown acid in your calculations instead of the correct volume of 20.00 mL. c. _____

d. Forgetting to rinse down the inside wall of the flask with deionized water during and at the end of the titration. d. _____

e. Reading the NaOH initial buret reading as 0.02 mL instead of the correct value of 1.02 mL. e. _____

3. Give a brief answer to each of the following. Write your answers in complete sentences in English.
- Why must the containers in which you store your unknown acid solution and your standard NaOH solution be completely dry before you add the solutions to them?
 - Why should you NOT plan to start the titration with the acid and base burets filled exactly to the zero mark?
 - Why should you titrate into an Erlenmeyer flask rather than into a beaker?