STADARDIZATION OF A BASE
Experiment #4

PURPOSE:

- To titrate a standard hydrochloric acid solution.
- To determine the exact molarity of a sodium hydroxide solution

PRINCIPLES:

Titration is a convenient volumetric method for accurately determining the concentration of solutions. In general, the process consists of the careful addition of a solution of one reagent whose concentration is to be determined to a solution of another reagent of exactly known concentration until the reaction between these substances is complete. The solution of the reagent of exactly known concentration is called a standard solution and the measured volume of the other solution can be used in calculations to determine the unknown concentration.

A crucial part of a successful titration is that a chemical reaction takes place. However, not all chemical reactions are equally suitable for this type of volumetric analysis. The most successful titrations involve reactions that meet or closely approach the following requirements:

1. The reaction should be rapid, so that the titration is not too slow and tedious.
2. The reaction should be stoichiometric, i.e., capable of being described by a chemical equation so that the volumetric data can be used directly in the titration calculation.
3. The reaction should not involve alternate or side reactions between constituents of the solutions.
4. There must be a method for determining when the reaction is complete.

Several general types of reactions come to mind which often meet these criteria. These are:

1. Reactions between acids and bases
2. Oxidation-reduction reactions
3. Reactions involving the formation of complexes
4. Precipitation reactions

Only the first of these categories will be considered here.

In general, acid - base titrations are performed by adding controlled quantities of base solution from a buret to a flask containing an accurately known volume of an acid solution. The objective or the titration is to add a quantity or base which reacts completely with an exactly measured volume of acid.

In this experiment the concentration of a basic solution will be determined by measuring the volume of the base required to completely neutralize a precisely measured volume of an acid of known molarity. The acid is hydrochloric acid, HCl, which is an important component in stomach digestive juices. The base is sodium hydroxide, commonly known as lye. The reaction can be written as:

$$HCl_{(aq)} + NaOH_{(aq)} \rightarrow NaCl_{(aq)} + H_2O_{(l)}$$

or more simply:

$$H^+_{(aq)} + OH^-_{(aq)} \rightarrow H_2O_{(l)}$$

Note that one mole of H^+ ions reacts exactly with one mole of OH^-. This implies in turn, that:

Number of moles of acid = Number of moles of base

or

(Volume of Acid) x (Molarity of acid) = (Volume of base) x (Molarity of Base)

$$\mathbf{V_{acid} \times M_{acid} = V_{base} \times M_{base}}$$

METHOD:

A solution of sodium hydroxide will be prepared by the dilution of a stock solution of sodium hydroxide. A solution of hydrochloric acid of known concentration will be titrated with a portion of the prepared sodium hydroxide solution using phenolphthalein as an indicator.

From the volume of the sodium hydroxide solution used and the volume and molarity of the hydrochloric acid solution, the molarity of the sodium hydroxide solution will be calculated.

PROCEDURE:

1. **Preparation of NaOH solution.**
    a. Measure 8.4 mL of 6 M sodium hydroxide solution in a 10-mL graduated cylinder.
    b. Pour the solution into a clean 500-mL plastic bottle.
    c. Dilute this solution to approximately 500 mL with deionized water. (The volume of the sodium hydroxide solution does not have to measured or known accurately, since this solution will be titrated in the next part of the experiment to determine its precise molarity.)
    d. Close the plastic bottle well.
    e. Mix the solution thoroughly by inverting the plastic bottle ten times. (Insufficient mixing of solutions is a common source of error in titrations.)
2. **Titration of the NaOH solution**
    a. Before the titration is begun, all glassware must be thoroughly cleaned. (The glassware usually needed for a titration includes a buret, a pipette, and an Erlenmeyer flask.)
    b. Obtain approximately 125 mL of standard 0.1 M HCl solution in a clean dry 250-mL or 200-mL beaker. (Your beaker must be dry or the water present in the beaker will dilute the hydrochloric acid solution and change the molarity of the solution.)
    c. Record the precise molarity of the hydrochloric acid solution.
    d. Rinse a clean 25-mL volumetric pipette with a 5-mL portion of deionized water.
        i. Always use a rubber bulb to draw water or solutions into pipette.
        ii. Make certain that the rinse water contacts the entire inner surface of the pipette.
        iii. Discard the rinse water.
        iv. Rinse the pipette at least two times with 5-mL portions of the standard HCl solution by holding the pipette in a horizontal position and rotating it so that the solution contacts the entire inner surface of the pipette.
        v. Discard the rinse solutions.

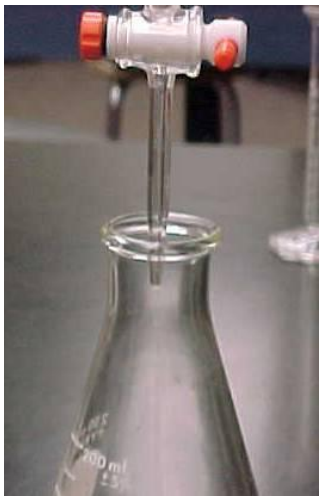
e. Carefully pipette 25.00 mL of the acid solution into the 250-mL Erlenmeyer flask.
    i. Hold the tip of the pipette against the inner surface of the flask to avoid splatter.
    ii. When the flow of liquid from the pipette stops, continue to hold the pipette in a vertical position for 15 seconds to allow reproducible draining of the pipette.
    iii. Touch off the last drop of solution on the tip of the pipette so that the drop enters the flask.
f. Add about 50 mL of deionized water to the acid solution in the flask to give sufficient volume of solution in which to see a color change.
    i. The addition of about 50 mL of deionized water to the 25.00 mL of hydrochloric acid will not affect the number of moles of HCl present in the solution, and will not affect the volume of titrant or number of moles of base to be added.
g. Add to the acid solution three drops of phenolphthalein solution and swirl the flask to thoroughly mix the solution.
h. After the buret has been properly cleaned, rinse the buret with a 5-mL portion of the 0.1 M NaOH solution so that the solution comes in contact with the entire inner surface of the buret and the tip of the buret.
    i. Drain the rinse solution through the tip of the buret and discard the rinse solution.
    ii. Repeat this procedure twice with two new 5-mL portions of the NaOH solution.
    iii. Discard the rinse solution through the tip of the buret.

NOTE: Since the sodium hydroxide solution tends to absorb carbon dioxide from the atmosphere, as shown in the equation below:

$$2NaOH_{(aq)} + CO_{2(g)} \rightarrow Na_2CO_{3(aq)} + H_2O_{(l)}$$

Because of this, ***the plastic bottle containing the sodium hydroxide solution should be always closed.*** If this precaution is not observed, the concentration of the solution is gradually lowered since the carbon dioxide absorption uses up some of the sodium hydroxide.

i. Close the stopcock and fill the buret with the solution of base to above the top calibration mark on the buret. After eliminating air bubbles in the tip of the buret, lower the meniscus of the solution until it is at a point on the calibrated portion of the buret.
j. Record the initial buret reading to the nearest 0.01 mL.



k. Place your 250 mL Erlenmeyer flask containing the acid solution under the buret and lower the buret tip until it is well into the mouth of the flask as shown.

l. Swirl the flask containing the acid solution with the right hand as the stopcock is controlled with the left hand.

Notes:

- If the standard HCl solution is approximately 0.1 M and the NaOH solution is also approximately 0.1 M then about 25 mL of NaOH solution should be require for the titration.
- If the concentration of the NaOH is correct, 18 to 20 mL of titrant may be added to the acid solution without danger of adding so much titrant that the end-point of the titration is exceeded.
- As the titration progresses, the approach of the end point will be signaled by a very temporary appearance of the end-point color where the titrant first comes in contact with the acid solution.
- As the end-point is approached more closely, these temporary flashes of color persist longer and smaller amounts of titrant should be added. Just prior to the end-point, titrant should be added one drop at a time. Fractions of a drop may even be added by allowing a droplet to begin to form on the buret tip and, after touching the buret tip to the inner surface of the flask, by washing down the inner surface of the flask with a stream of distilled water from a wash bottle.
- The titrant may splatter slightly as it is added from the buret. If this occurs, the inner surface of the Erlenmeyer flask should be washed down thoroughly with deionized water before the end-point is reached.
- The titration is complete when the first barely perceptible, but permanent end-point color appears. The best accuracy is obtained if the intensity of the pink color is the faintest that can be seen.
- Divide the last drop of titrant if possible.

m. Read the final buret volume to the nearest 0.01 mL and record this volume.

n. Repeat the titration at least two more times.

i. Titrations should be done until three results are obtained which agree to within three parts thousand.

ii. Between titrations, discard the neutralized solution from the Erlenmeyer flask. Rinse the flask several times with a few milliliters of deionized water.

iii. The flask need not be dried before introducing the 25.00 mL of hydrochloric acid for the next titration.

o. When all of the titrations are completed, drain and rinse the buret thoroughly with deionized water to remove all of the sodium hydroxide solution.

p. Rinse other glassware also in order to have it ready to use for the next determination.

CALCULATIONS:

1. For each titration, calculate the molarity of the sodium hydroxide solution.

$$M_{base,moles/L} = \frac{(M_{acid,moles/L})(V_{acid,Liters})}{V_{base,Liters}}$$

2. Calculate the average molarity of the sodium hydroxide solution.

3. Calculate **The Relative Mean Deviation** (RMD) of your base molarity:

$$RMD = \frac{Mean\ Deviation}{Mean\ Molarity}\ x\ 1000 = \frac{\overline{d}}{\overline{X}}\ x\ 1000\ (parts\ \ per\ thousand)$$

$$\overline{d} = mean\ deviation = \frac{\sum \left| X_i - \overline{X} \right|}{n}$$

$\overline{X}$ = mean molarity

X_i = individual molarity value

n = number of results

GOOD STUDENT WORK SHOULD HAVE AN RMD OF LESS THAN 3 PARTS PER THOUSAND.

EXPERIMENT #4
REPORT FORM

Molarity of Standard HCl solution: _____M

Volume of Standard HCl solution used: 25.00 mL

	TITRATIONS		
	First	Second	Third
Final buret reading (mL)	_____	_____	_____
Initial buret reading (mL)	_____	_____	_____
Volume of titrant (mL)	_____	_____	_____
Moles of HCl (mol)	_____	_____	_____
Moles of NaOH (mol)	_____	_____	_____
Molarity of NaOH (M)	_____	_____	_____

Show one sample calculations below:

Average molarity of NaOH solution (M) _____

Deviation $|d_i|$ _____ _____ _____

Mean deviation _____

Relative Mean Deviation, RMD _____

NOTE: Good student work should have an RMD of less than 3 parts per thousand.