Purpose

To quantitatively determine the concentration of an acid using an indicator that detects the equivalence point in a neutralization reaction.

Learning Objectives

Explore acid–base chemistry.

Standardize an acid solution.

Learn how to use indicators in titrations.

Determine the acidity level of vinegar.

Reinforce using volumetric methods for quantitative analysis.

Discussion

Acids and bases play central roles in living and environmental systems. Household items include citrus fruits like lemons and oranges (acids), vinegar (acid), baking soda (base), and drain and toilet bowl cleaners (bases). The pH scale expresses acidity, with pH < 7 representing an acidic solution and pH > 7 representing a basic solution. You may be familiar with "pH-balanced" shampoos. The body maintains the pH of blood within a very narrow range in a healthy individual, and deviations lead to disease conditions. When the acidity in streams increases above a threshold level, fish and aquatic life die. Acid rain produces serious consequences. However, the environmental controls on the emission of acidic substances (especially SO_2, which converts to sulfuric acid in the atmosphere) from coal-fired electric power plants has greatly reduced the harmful effects of acid rain over the last 20 years.

Part 1. Standardization of Sodium Hydroxide Solution

This part of the experiment reinforces the **volumetric** method of **quantitative analysis**. Volumetric methods involve determining the amount of one substance that reacts with another by measuring a volume. The key to using a volume in a quantitative determination requires knowing the concentration accurately, because calculating the number of moles requires both the volume and concentration of solute that have reacted.

In a typical volumetric method, a measured volume of one solution of known concentration is added to another solution containing the substance whose concentration is to be determined. The experiment is set up so that some property of the solution (often a change in its color) results when an **equivalent** amount of the solution of known concentration has been added. This procedure is called a **titration**, and the point at which the observation

occurs is called the **endpoint**. Because water and many typical reactants used in titrations are colorless, a wide range of different substances called **indicators** have been developed. The indicator changes colors when the endpoint is reached, without influencing the stoichiometric reaction being studied. For this reason, indicators are usually very strongly colored species that will be clearly visible to the eye at very small concentrations.

A **standard solution** is necessary to carry out a volumetric analysis. A standard solution is one whose concentration is accurately known. This concentration may, in turn, be determined by titration of a solution containing a known mass of a **primary standard**. A primary standard is often a substance obtained in high purity, dried to constant weight, and does not undergo any side reactions during the titration. In this experiment, students will use a primary standard previously prepared to work within the time constraints of the lab.

A standard solution of sodium hydroxide (NaOH) cannot be prepared by weighing out a sample and dissolving it in an accurately measured volume of water. Solid NaOH absorbs water quickly from the air (**hygroscopic**), making it impossible to accurately know the weight due to NaOH and the weight due to water. A NaOH solution must, therefore, be titrated with a primary standard to determine its concentration. The standard used in this experiment is a hydrochloric acid (HCl) solution of known molarity.

In this experiment, students will quantitatively study an acid–base reaction. Strong acids and strong bases dissociate completely in water as depicted in Reaction AB.1 and Reaction AB.2.

$$HCl + H_2O \longrightarrow H_3O^+ + Cl^- \qquad \text{(Reaction AB.1)}$$

$$NaOH \longrightarrow Na^+ + OH- \qquad \text{(Reaction AB.2)}$$

So the reaction of a strong acid and a strong base in aqueous solution is simply as shown in Reaction AB.3:

$$H_3O^+ + OH^- \longrightarrow 2\,H_2O \qquad \text{(Reaction AB.3)}$$

Recall that this reaction is called **neutralization**. The sodium and chloride ions remain unchanged in solution and are therefore termed **spectator ions**.

At the equivalence point, the number of moles of added base is stoichiometrically equivalent to the moles of acid being titrated. Note that the ratio of the reactants in the above acid–base reaction is 1 mol HCl per 1 mol NaOH. This is a 1:1 reaction stoichiometry. When the HCl is exactly neutralized by the NaOH, it follows that the moles of acid (n_A) and base (n_B) are equal. Thus, at the equivalence point of the titration, the number of moles of NaOH added from the buret must equal the number of moles of HCl originally present in the flask. The moles of HCl in the solution is found using Equation AB.1:

$$M_{HCl} \times V_{HCl} = n_{HCl} \qquad \text{(Equation AB.1)}$$

In Equation AB.1, V_{HCl} is the volume of standard HCl titrated. Students will measure the volume of NaOH solution necessary to reach the equivalence point in the reaction between HCl and NaOH. Knowing the volume of NaOH added from the buret and the moles of added NaOH, the molarity of NaOH can be found using Equation AB.2 and Equation AB.3:

$$n_{HCl} = n_{NaOH} \qquad \text{(Equation AB.2)}$$

$$M_{NaOH} = \frac{\text{moles NaOH}}{\text{volume (L) NaOH}} = \frac{n_{NaOH}}{V_{NaOH}} \qquad \text{(Equation AB.3)}$$

All acid–base indicators are weak acids designated by the formula HA (A is usually a complicated organic molecule). For clarity, indicators are often written as H-Ind, and their conjugate base is written as Ind^-. The use of "Ind" for indicator distinguishes the indicator, which is a weak acid, from the weak acid that might be something one is trying to analyze. The function of an indicator results from the fact that the acid form and the conjugate base form have distinctly different colors. In the case of phenolphthalein, the acid form is colorless and the basic form is bright pink. Figure AB.1 depicts the structures of phenolphthalein. Note that phenolphthalein, under ordinary conditions, is a **diprotic acid**. Actually two protons are removed to afford the basic (pink) form. For this reason, it is more appropriate to use the label H_2Ind and Ind^{2-} for these species.

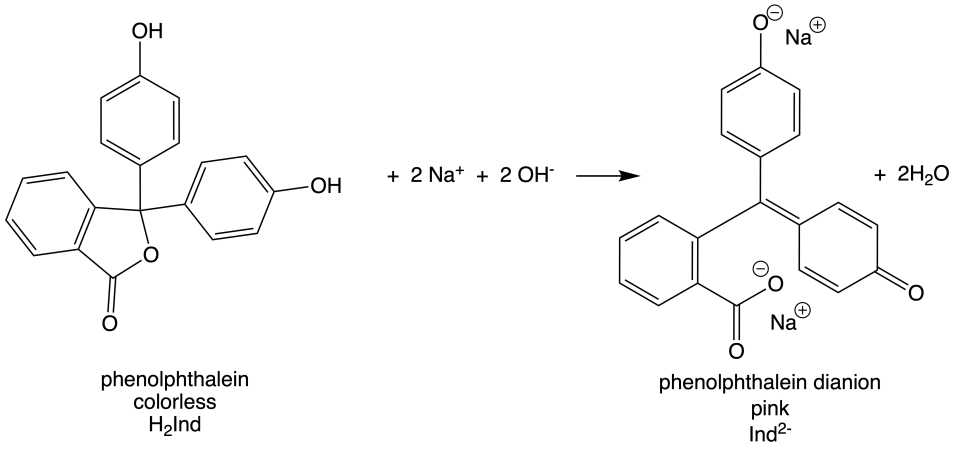

Figure AB.1: Reaction of phenolphthalein with sodium hydroxide

When a solution is acidic, the indicator will be present in its acid form (H_2Ind). However, at the point that enough NaOH has been added to neutralize all of the acetic acid, the indicator will begin to react. At that point, the addition of even a tiny amount of additional NaOH will convert the acid form of phenolphthalein to the basic, colored form (Ind^{2-}), because the indicator concentration is so small. This feature is important because it allows the endpoint to be both clearly visualized and accurate.

Part 2. Determination of the Acetic Acid (CH_3COOH) Content of Vinegar

Vinegar is a dilute solution of acetic acid, $HC_2H_3O_2$, an organic acid. The structure of acetic acid may be represented as CH_3CO_2H or CH_3COOH. In Figure AB.2, the dashed circle indicates the acidic hydrogen.

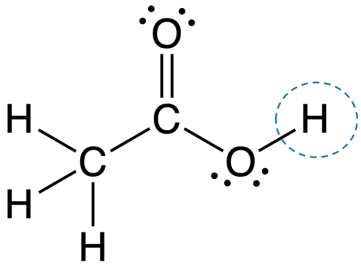

Figure AB.2: Molecular structure of acetic acid. Circle indicates the acidic hydrogen.

The COOH portion of the molecule is called a carboxylic acid functional group, and it accounts for the acidic behavior. Because there is only one acidic hydrogen in acetic acid, it is a **monoprotic acid**. Titration with a strongly basic solution of known concentration can determine the acidic content of a vinegar sample.

A weak acid is one that dissociates only slightly. When a weak acid is reacted with a strong base, the base is removing a hydrogen ion (we often say "proton") from a dissociated acid molecule depicted in Reaction AB.4.

$$HA + OH^- \longrightarrow H_2O + A^- \qquad \text{(Reaction AB.4)}$$

In Reaction AB.4, HA represents the weak acid and A^- is its conjugate base (removing H^+ from HA leaves A^-). In the main part of your experiment, HA is CH_3COOH (acetic acid) and A^- is CH_3COO^- (acetate anion).

Procedure

Safety Precautions

Hydrochloric Acid: Corrosive

Sodium Hydroxide: Corrosive

Phenolphthalein: Irritant and Flammable

Vinegar Solution: Irritant

Part 1. Standardization of Sodium Hydroxide Solution.

1. Use a 250-mL beaker (not a graduated cylinder) to obtain approximately 75 mL of standard HCl solution and use a 400-mL beaker (not a graduated cylinder) to obtain approximately 125 mL of NaOH solution. Record the molarity of the primary acid solution from the bottle label.

2. Wash the burets before using them (rinse with distilled water). Use a funnel to fill one buret with the HCl solution and the second buret with the NaOH solution. Rinse funnel between fillings.

Safety Precautions

Caution: The solutions are corrosive. In case of accidental skin contact, wash with plenty of water.

3. Open the stopcock on each buret and let a small amount of solution flow out of the buret into a waste beaker to ensure that the buret tip is filled with solution.

4. Dispense 25.00 mL of standard HCl solution from the buret into a clean (not necessarily dry) 250-mL Erlenmeyer flask (not a beaker) and add two drops of phenolphthalein solution.

> DO NOT refill the burets between titrations. The final reading from measurement 1 is the initial reading for measurement 2. If a third measurement is required, only fill the buret with the approximate amount of solutions needed.

5. Titrate the HCl with the NaOH solution until the pink color of the indicator persists for at least 1 minute. Record the volume of the NaOH solution used in a table similar to Table AB.1.

Table AB.1: Buret Data for NaOH Standardization

Trial	Initial Buret Reading for NaOH	Final Buret Reading for NaOH
1	_____	_____
2	_____	_____
3	_____	_____

6. Titrate a second 25.00 mL sample of the standard acid against the NaOH solution, as above. If the two values are more than 0.2 mL apart perform a third titration and discuss the results with your instructor before proceeding further.

7. Pour the liquid waste into the liquid waste container. Do not pour down the sink.

Part 2. Determination of the Acetic Acid (CH_3COOH) Content of Vinegar

1. Take a clean, dry 50-mL beaker and obtain approximately 30 mL of the vinegar solution.

2. Wash and rinse your 25.00-mL serological pipet with water. Next, rinse using a small amount of the vinegar solution, and then pipet 25.00 mL of the vinegar to a clean 250-mL volumetric flask.

3. Dilute the concentrated vinegar to the mark with distilled water, stopper the flask, swirl and invert to mix thoroughly. Transfer the solution to a clean, dry beaker to make pipetting easier. Remember to rinse the pipet again because it will contain traces of the concentrated vinegar.

4. Titrate three 25.0-mL aliquots of this solution with the now known standardized NaOH solution using phenolphthalein as an indicator. Record your results.

> DO NOT refill the burets between titrations. The final reading from measurement 1 is the initial reading for measurement 2. Similarly, the final reading from measurement 2 is the initial reading for measurement 3

Table AB.2: Initial and Final Buret Readings (Acetic Acid Titration)

Trial	Initial Buret Reading for NaOH	Final Buret Reading for NaOH
1	_____	_____
2	_____	_____
3	_____	_____

5. Dispose of the liquid waste into the liquid waste container in the hood.

6. Return the buret to buret holder and hang upside down to dry.

Supplemental Equations

Below provides the equations needed to solve the required calculations. Several of these may be combined to shorten the number of equations needed; students can determine the best approach and number of steps that best suits their approach to determining the required calculations.

$$n_{\text{NaOH}}\,(\text{mol}) = n_{\text{HCl}}\,(\text{mol})$$

$$M_{\text{NaOH}}\left(\frac{\text{mol}}{\text{L}}\right) = \frac{M_{\text{HCl}}\left(\frac{\text{mol}}{\text{L}}\right) \times V_{\text{HCl}}\,(\text{L})}{V_{\text{NaOH}}\,(\text{L})}$$

$$n_{\text{NaOH}}\,(\text{mol}) = M_{\text{NaOH}}\left(\frac{\text{mol}}{\text{L}}\right) \times V_{\text{NaOH}}\,(\text{L})$$

$$n_{\text{dilute vinegar}}\,(\text{mol}) = n_{\text{NaOH}}\,(\text{mol})$$

$$M_{\text{dilute vinegar}}\left(\frac{\text{mol}}{\text{L}}\right) = \frac{n_{\text{dilute vinegar}}\,(\text{mol})}{V_{\text{dilute vinegar}}\,(\text{L})}$$

$$M_{\text{dilute vinegar}}\left(\frac{\text{mol}}{\text{L}}\right) = \frac{M_{\text{NaOH}}\left(\frac{\text{mol}}{\text{L}}\right) \times V_{\text{NaOH}}\,(\text{L})}{V_{\text{dilute vinegar}}\,(\text{L})}$$

$$M_{\text{stock vinegar}} = M_{\text{undiluted vinegar}} \times \text{Dilution factor}$$

$$M_{\text{stock vinegar}}\left(\frac{\text{mol}}{\text{L}}\right) \times V_{\text{stock vinegar}}\,(\text{L}) = M_{\text{diluted vinegar}}\left(\frac{\text{mol}}{\text{L}}\right) \times V_{\text{diluted vinegar}}\,(\text{L})$$

$$M_{\text{stock vinegar}}\left(\frac{\text{mol}}{\text{L}}\right) = \frac{M_{\text{diluted vinegar}}\left(\frac{\text{mol}}{\text{L}}\right) \times V_{\text{diluted vinegar}}\,(\text{L})}{V_{\text{stock vinegar}}\,(\text{L})}$$

$$n_{\text{acetic acid in stock vinegar}}\,(\text{mol}) = M_{\text{stock vinegar}}\left(\frac{\text{mol}}{\text{L}}\right) \times V_{\text{stock vinegar}}\,(\text{L})$$

$$\text{Mass}_{\text{acetic acid}}(\text{g}) = n_{\text{acetic acid in stock vinegar}}\,(\text{mol}) \times \text{molar mass}_{\text{acetic acid}}\,(\text{g/mol})$$

$$\text{Mass \%} = \frac{\text{mass}_{\text{acetic acid}}\,(\text{g})}{\text{mass}_{\text{vinegar}}\,(\text{g})} \times 100 = \frac{\text{mass}_{\text{acetic acid}}\,(\text{g})}{V_{\text{stock vinegar}}\,(\text{L}) \times \text{density}_{\text{vinegar}}\left(\frac{\text{g}}{\text{L}}\right)} \times 100$$