Experiment 3
STOCHIOMETRY OF SOLUTIONS

INTRODUCTION

A. Acid-Base Reaction: Analysis of Acetic Acid in Vinegar

Neutralization is the reaction between an acid and a base. The important feature of this reaction in aqueous solution is the combination of hydronium ion with hydroxide ion to form water: $H_3O^+ + OH^- \rightarrow 2H_2O$. In a neutralization reaction, a point is reached where both acid and base are consumed and neither is in excess. This is called the equivalence point of neutralization.

To locate the equivalence point in a neutralization reaction requires that careful control be exercised over the addition of a base to an acid (or acid to base) and a means be found to signal the point at which the reaction mixture turns from acid to base (or base to acid). The first objective can be achieved by dispensing one solution into the other through a burette, a long tube with volume markings and a stopcock at the bottom. This device measures accurately the volumes of solutions used. The second objective is achieved by using an acid-base indicator, a substance that, if properly chosen, changes color when the solution changes from having a very slight excess of acid to having a very slight excess of base. This analytical technique of accurately measuring the volume of a solution required to react with another solution is termed titration. The solution of known concentration is the titrant (or standard solution) and the solution of unknown concentration is the analyte. The point at which the indicator changes color is the endpoint of titration. The endpoint of titration can differ slightly from the equivalence point.

In this experiment, the acidity of several brands of vinegar is to be determined by titrating it with standard sodium hydroxide solution using phenolphthalein as indicator. Vinegar is a solution that is 4-6 percent by weight acetic acid in water. Generally, caramel flavoring and coloring are added to make the product sell better. The chemical equation involved is:

$$HC_2H_3O_2 + NaOH \rightarrow NaC_2H_3O_2 + H_2O$$

B. Redox Reaction: Analysis of Dissolved Oxygen in Tap Water

The oxygen normally dissolved in water is important to fish and other aquatic organisms. Certain pollutants deplete the dissolved oxygen during the course of their decomposition. This is true for many organic compounds that are present in sewage or dead algae. These are decomposed by microorganisms, which use these organic compounds for food. The metabolic process is the oxidation of the organic compounds—the dissolved oxygen is the oxidizing agent. Thus while these microorganisms are removing the pollutants, they are also removing the dissolved oxygen that otherwise would be present to support aquatic life. Since the solubility of gases in solution decreases as the temperature of the solution increases, thermal pollution also decreases the dissolved oxygen content. Lost dissolved oxygen can be replenished as atmospheric oxygen enters during mixing in the form of waves. When the rate of oxygen usage exceeds the rate at which the oxygen can be supplied, the body of water may become anaerobic. As a logical consequence of this, one empirical standard for determining water quality is the dissolved-oxygen content (DO). The survival of aquatic life depends upon the ability of water to maintain certain minimum concentrations of the vital dissolved oxygen. At normal temperature, the maximum amount of oxygen that can possibly dissolve in water is about 9 mg/L (or 9 ppm). For a diversified warm-water biota, the DO concentration should be at least 5ppm.

In this experiment, the amount of dissolved oxygen in a water sample is to be determined by using the iodometric (or Winkler) method. This procedure is based on the use of manganous compounds that are oxidized to manganic compounds by the oxygen in the water sample. The manganic compound in turn reacts with potassium iodide to produce iodine. The released iodine is then titrated with standardized sodium thiosulfate using starch as the indicator. The chemical reactions involved are as follows:

$$MnSO_4 + 2NaOH \rightarrow Mn(OH)_2(s) + Na_2SO_4$$
$$2Mn(OH)_2(s) + O_2 \rightarrow 2MnO(OH)_2(s)$$
$$MnO(OH)_2(s) + 2H_2SO_4 \rightarrow Mn(SO_4)_2 + 3H_2O$$
$$Mn(SO_4)_2 + 2KI \rightarrow MnSO_4 + K_2SO_4 + I_2$$
$$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$$

The net overall chemical equation for this sequence of reactions is:

$$\tfrac{1}{2}O_2 + 2S_2O_3^{2-} + 2H^+ \rightarrow S_4O_6^{2-} + H_2O$$

OBJECTIVES

1. To compute the percent by weight of acetic acid in a vinegar sample
2. To compute the dissolved oxygen content of a water sample

LIST OF CHEMICALS

65 mL	0.2M sodium hydroxide	10 mL	vinegar samples
6 drops	phenolphthalein	6 mL	1% starch solution
65 mL	0.025M sodium thiosulfate	10 mL	concentrated sulfuric acid
15 mL	1M hydrochloric acid		
10 mL	manganous sulfate solution (450 g $MnSO_4 \bullet 4H_2O$ per L solution)		
10 mL	alkaline iodide reagent (360 g NaOH and 200 g KI per L solution)		

LIST OF APPARATUS

base burette, burette clamp, iron stand, two 150-mL beaker, 10- mL graduated cylinder, 125-mL Erlenmeyer flask, 3 measuring pipettes, acid burette, 100-mL graduated cylinder, 500-mL Erlenmeyer flask, 500-mL plastic container, rubber bulb

PROCEDURE

I. Acid-Base Reaction: Analysis of Acetic Acid in Vinegar

A. Preparation of a Burette for Use

1. Clean a 25-mL base burette with a soap solution and a burette brush and thoroughly rinse it with tap water.

2. Then rinse with 10-mL portion of distilled water. Do the rinsing for 3 times. The water must run freely from the burette without leaving any drop adhering to the side.

3. Make sure that the burette does not leak. Tell your instructor if your burette is leaking.

B. Analysis of Acetic Acid in Vinegar

Note: Each group will use a different brand of vinegar.

1. Rinse the previously cleaned base burette with approximately 5 mL of 0.2M sodium hydroxide solution (standard solution), making certain that no water droplet clings to the inside wall of the burette. Allow some of the solution to run out through the tip of the burette. Discard the sodium hydroxide solution used in a beaker. Repeat the rinsing process two more times.

2. Clamp the burette to an iron stand. See Figure 1.

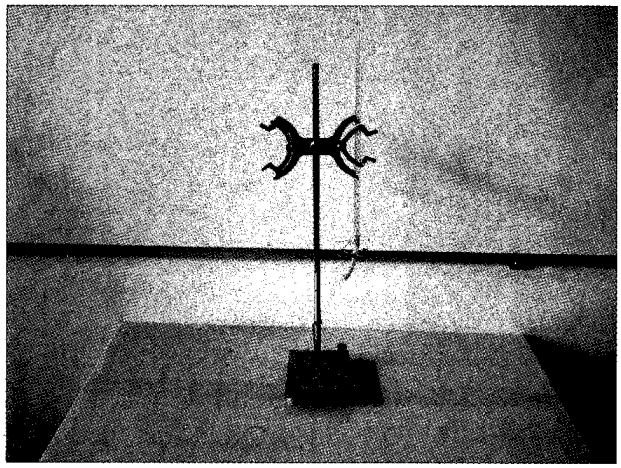

Figure 1. Titration set-up

3. Completely fill the burette with the 0.2M sodium hydroxide solution and remove the air from the tip by running out some of the solution into the beaker used for the washings.

4. Make sure that the lower part of the meniscus is at the zero mark.

5. Remove any hanging drop from the burette tip by touching it to the side of the beaker used for the washings.

6. Measure 5 mL of vinegar using a 10-mL graduated cylinder and place it in a 125-mL Erlenmeyer flask.

7. Add 20 mL of distilled water and 2 drops of phenolphthalein. Mix the solution by swirling.

8. Add slowly the sodium hydroxide solution while gently swirling the contents of the flask. Stop the titration when the color of the vinegar changes to light pink. See Figure 2.

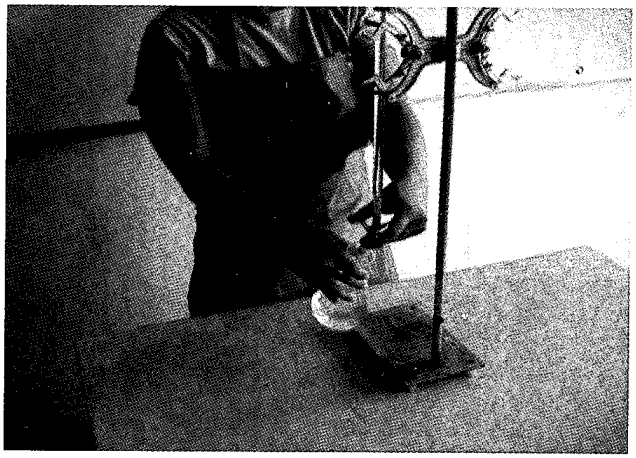

Figure 2. Titration of vinegar

Note: As the sodium hydroxide solution is added, a pink color appears where the drops of the base come in contact with the vinegar. This color disappears with swirling. As the endpoint is reached, the color disappears more slowly, at which time the sodium hydroxide solution should be added drop by drop. It is important that the flask be swirled constantly throughout the entire titration. The endpoint is reached when one drop of the sodium hydroxide solution turns the entire vinegar in the flask from colorless to light pink. The vinegar should remain light pink when it is swirled. A sheet of white paper underneath the flask will help in recognizing the color change at the endpoint.

9. Record the volume of the sodium hydroxide solution used in the titration.

10. Make another trial.

11. **Waste Disposal:** The titrated solution can be disposed to the sink. Add 2 drops of phenolphthalein to the sodium hydroxide used in washing the burette. Neutralize it by adding dropwise 1M hydrochloric acid until the color turns light pink. Dispose to the sink.

C. Computation of the Percent by Weight of Acetic Acid in the Vinegar

1. Compute the moles of sodium hydroxide used in the titration.

Moles NaOH = (volume NaOH in liter) X (molarity NaOH in moles/liter)

2. Calculate the moles of acetic acid in the vinegar.

$$\text{Moles } HC_2H_3O_2 = (\text{moles NaOH}) \times \frac{1 \text{ mole } HC_2H_3O_2}{1 \text{ mole NaOH}}$$

From the balanced chemical equation given in the introduction of this experiment, the molar ratio of acetic acid to sodium hydroxide is 1:1.

3. Determine the weight of acetic acid in the vinegar.

$$\text{Weight } HC_2H_3O_2 = (\text{moles } HC_2H_3O_2) \times (\text{molar weight of } HC_2H_3O_2)$$

The molar weight of acetic acid is 60 g/mole.

4. Compute the weight of the vinegar.

$$\text{Weight of vinegar} = (\text{volume of vinegar}) \times (\text{density of vinegar})$$

Assume that the density of vinegar is 1 g/mL.

5. Calculate the percent by weight of acetic acid in the vinegar.

$$\%\text{ by weight } HC_2H_3O_2 = \frac{\text{weight } HC_2H_3O_2}{\text{weight of vinegar}} \times 100$$

6. Compute the average percent by weight of acetic acid in the vinegar.

II. Redox Reaction: Analysis of Dissolved Oxygen in Tap water

A. Collection of Water Sample (Tap Water)

1. Open the faucet (but not fully opened) and let the water run for about one minute.

2. Collect the water sample in a clean 500-mL plastic container with cap. The water sample must be taken with extreme care to minimize contact of the sample with air. It is therefore desirable to let the water run down the sides of the container. See Figure 3.

3. Allow the container to overflow for about a minute keeping the opening of the faucet below the water level and replace the cap so that no air bubbles are entrained. See Figure 4. Avoid excessive agitation that will dissolve

atmospheric oxygen. The water sample should be analyzed as soon as possible.

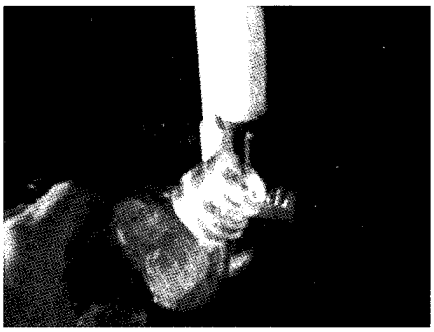

Figure 3. Collecting sample

Figure 4. Overflowing the container

B. Release of Iodine

SAFETY PRECAUTION: All chemical reagents to be used are corrosive. If you spill any of these reagents on your skin, immediately rinse the affected area with water. Handle them with care.

1. Open the sample container with great care to avoid aeration and add 5 mL of manganous sulfate solution from a measuring pipette. This is done by dipping the end of the pipette halfway of the water depth and releasing the contents of the pipette, causing an overflow of the water sample.

2. Similarly, add 5 mL of alkaline iodide reagent using another measuring pipette. The neck of the container will again have excess liquid so replace carefully the cap to avoid splashing.

3. Mix thoroughly the contents by making 2 rapid inversions of the container in the hand. A milky precipitate forms that gradually changes to yellowish-brown.

4. Allow the precipitate to settle.

5. When the precipitate has settled, remove carefully the cap and immediately add 5 mL of concentrated sulfuric acid in the same manner as before.

6. Close immediately the container and then mix the contents by gentle inversion until the precipitate has completely dissolved. At this point, the yellowish-brown color due to liberated iodine should appear.

C. Titration

1. Rinse a clean base burette with approximately 5 mL of 0.025M sodium thiosulfate solution (standard solution), making certain that no water droplet clings to the inside wall of the burette. Allow some of the liquid to run out through the tip of the burette. Discard the sodium thiosulfate solution used to the sink with plenty of water. Repeat the rinsing process two more times.

2. Fill the cleaned 25-mL base burette with 0.025M sodium thiosulfate solution, making sure that there is no air gap at the tip of the burette.

3. Attach the burette to an iron stand using a burette clamp.

4. Using a 100-mL graduated cylinder, measure 200 mL of the sample and place it in a 500-mL Erlenmeyer flask.

5. Add 3 mL of 1% starch solution.

6. Titrate the sample with the standard sodium thiosulfate solution until the color changes from blue to colorless. Always swirl the flask while adding sodium thiosulfate.

7. Record the volume of the standard sodium thiosulfate solution used.

8. Make 2 trials.

9. **Waste Disposal:** Dispose all solutions to the sink with plenty of water.

D. Calculation of Dissolved Oxygen Content

1. Calculate the moles of sodium thiosulfate used in the titration of the 200-mL portion of the water sample.

Moles $Na_2S_2O_3$ = (volume $Na_2S_2O_3$ in liter) X (molarity $Na_2S_2O_3$ in moles/liter)

2. Compute the moles of dissolved oxygen.

$$\text{Moles } O_2 = (\text{moles } Na_2S_2O_3) \times \frac{\frac{1}{2} \text{ mole } O_2}{2 \text{ moles } Na_2S_2O_3}$$

From the net overall reaction given in the introduction of this experiment, the molar ratio of oxygen to sodium thiosulfate is ½ : 2.

3. Determine the weight of dissolved oxygen.

$$\text{Weight } O_2 = (\text{moles } O_2) \times (\text{molar weight of } O_2)$$

The molar weight of oxygen is 32 g/mole.

4. Calculate the dissolved oxygen content of the water sample in ppm.

$$\text{ppm } O_2 = \frac{\text{milligrams } O_2}{\text{Liter sample}}$$

5. Compute the average dissolved oxygen content of the water sample in ppm.

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Group No._____

Name:	**Date Performed:**
Course & Section:	**Date Submitted:**
Program & Year:	**Professor:**

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STOICHIOMETRY OF SOLUTIONS

I. Acid-Base Reaction: Analysis of Acetic Acid in Vinegar

Brand of Vinegar:		
	Trial 1	Trial 2
Volume of 0.2M NaOH in mL		
Moles NaOH		
Moles $HC_2H_3O_2$		
Weight $HC_2H_3O_2$ in Grams		
Volume of Vinegar in mL		
Weight of Vinegar in Grams		
% by Weight $HC_2H_3O_2$		
Average % by Weight $HC_2H_3O_2$		

Show the computations for the following using the data in trial 1:

1. Moles NaOH

2. Moles $HC_2H_3O_2$

3. Weight $HC_2H_3O_2$

4. Weight of Vinegar

5. Percent by Weight $HC_2H_3O_2$

6. Average Percent by Weight $HC_2H_3O_2$

Comparison of the Acidity of the Different Brands of Vinegar

Group No.	Brand of Vinegar	% by Weight $HC_2H_3O_2$
1		
2		
3		
4		
5		
6		
7		
8		

Which vinegar is the most acidic? Why?

II. Redox Reaction: Analysis of Dissolved Oxygen in Tap water

	Trial 1	Trial 2
Volume of Water Sample in mL		
Volume of 0.025M $Na_2S_2O_3$ in mL		
Moles $Na_2S_2O_3$		
Moles O_2		
Weight of O_2 in Grams		
Dissolved Oxygen Content in ppm		
Average DO Content in ppm		

Show the calculations for the following using the data in trial 1:

1. Moles $Na_2S_2O_3$

2. Moles O_2

3. Weight of O_2

4. ppm Dissolved Oxygen

5. Average ppm of Dissolved Oxygen

Describe all the results observed as the different reagents used in the analysis are added:

1. manganous sulfate solution and alkaline iodide reagent

2. concentrated sulfuric acid

3. starch solution

4. sodium thiosulfate

Does the water sample contain sufficient dissolved oxygen to sustain aquatic life? Explain.