EXPERIMENT A7: VINEGAR TITRATION

Learning Outcomes

Upon completion of this lab, the student will be able to:

1) Prepare a solution of primary standard
2) Determine the molar concentration of a solution of an acid or base using data obtained from titration
3) Measure the amount of acetic acid in a solution of vinegar

Introduction

The molar concentration of an acid or a base can be determined by the method of titration. In a titration, a solution of known concentration is slowly added to a known volume of a solution of unknown concentration until the two have completely reacted with each other. There are many different kinds of titration; the type used in this experiment is called an acid-base titration.

Consider for instance, the reaction between aqueous solutions of sulfuric acid and potassium hydroxide. The balanced chemical equation for the reaction is shown below:

$H_2SO_{4(aq)} + 2KOH_{(aq)} \rightarrow K_2SO_{4(aq)} + 2H_2O_{(l)}$

Assume that the molar concentration of KOH is 0.100 M and that of H_2SO_4 is unknown. In order to determine the molar concentration of H_2SO_4, one would need to add the KOH solution to a known volume of H_2SO_4.

In a certain experiment, the KOH was added to 10.00 mL of H_2SO_4. The volume of KOH needed to completely react with the H_2SO_4 will enable the determination of the molarity of H_2SO_4. In the same experiment, assume that 13.75 mL of KOH was needed to completely react with the H_2SO_4. The molarity of the acid is calculated as follows:

Molarity of H_2SO_4 =

$$0.100\frac{mol}{L}KOH \times 13.75ml \times \frac{1L}{1000mL} \times \frac{1H_2SO_4}{2KOH} \times \frac{1}{10.00mL} \times \frac{1000mL}{1L} = 0.0688\frac{mol}{L}$$

As seen from the above calculation, the stoichiometric ratio between the two reactants is the key to the determination of the molarity of the unknown solution.

In order to conduct the above experiment, typically the H_2SO_4 is in an Erlenmeyer flask, and the KOH is in a burette. The KOH is added one drop at a time from the

burette into the acid solution with constant stirring to ensure that the reagents combine and react.

Vinegar Titration

In this experiment, a solution of vinegar has been provided for analysis. The active ingredient in vinegar is acetic acid, CH_3COOH. In order to determine the amount of acetic acid in the vinegar, the acetic acid will be titrated with a solution of known concentration of sodium hydroxide.

The chemical reaction between acetic acid and sodium hydroxide is given below:

$$CH_3COOH_{(aq)} + NaOH_{(aq)} \rightarrow CH_3COONa_{(aq)} + H_2O_{(l)}$$

The balanced chemical equation shows that one mole of CH_3COOH reacts with exactly one mole of NaOH. The experiment is performed by adding NaOH of known molarity to a known volume of vinegar until the reaction is complete.

Determining the completion of an acid/base reaction

In order to obtain the molarity or moles of the unknown reactant the solution whose concentration is known (in this experiment that would be NaOH), must be added until the reaction is complete. This means, exactly one mole of NaOH must be added to one mole of acetic acid. This point in the titration is called the **Equivalence Point**.

The equivalence point is defined as that point in the titration when stoichiometrically equal amounts of acid and base are present. In the CH_3COOH/NaOH titration, that would be when one mole of NaOH has been added to one mole of CH_3COOH. In the H_2SO_4/KOH example shown previously, that would be when two moles of KOH have been added to one mole of H_2SO_4. Therefore the equivalence point depends on the reaction stoichiometry.

At the beginning of the titration, the solution in the Erlenmeyer flask is acidic. As the base is added, it completely reacts with the acid and the solution in the Erlenmeyer flask continues to be acidic. But, at the equivalence point, the acid has completely reacted with the base. If even one tiny drop of base is added beyond that needed to arrive at the equivalence point, the solution in the Erlenmeyer flask is basic. This difference in the acid/base property of the solution in the Erlenmeyer flask is used to visually determine the end of the titration.

An **indicator** is a chemical substance whose color depends on the acid/base property of the medium it is present in. Phenolphthalein is an indicator, which is colorless in an acidic medium and has a pink color in a basic medium.

In this titration, a few drops of phenolphthalein should be added to the acid in the Erlenmeyer flask. The solution will remain colorless until the equivalence point.

When the equivalence point has been crossed and the solution becomes basic, the phenolphthalein will take on a pink color. This is the reason to add the base drop by drop, so that even though the equivalence point will be crossed, the titration can be stopped at the appearance of the first permanent pale pink color.

This point in the titration when the indicator changes color is referred to as the **End Point**. It is important to note that the End Point of a titration is slightly beyond the Equivalence Point. In the case of the phenolphthalein, the intensity of the pink color increases as the solution becomes more and more basic. Therefore, in order to stay as close to the Equivalence Point as possible, it is important to stop the titration at the appearance of a permanent pale pink color. In order to easily observe the color changes in the solution, it is a good idea to place a sheet of plain white paper beneath the flask.

Molarity of the sodium hydroxide solution

The data from the titration described in the previous sections can be used to determine the moles of acetic acid present in the vinegar solution. The accuracy of the calculation depends on knowing the molarity of the sodium hydroxide accurately. However, sodium hydroxide is an extremely hygroscopic solid. When solid sodium hydroxide is weighed using an electronic balance it tends to absorb moisture from the atmosphere; this leads to inaccuracies in the mass of the sodium hydroxide and thereby inaccuracies in its molarity calculations.

In order to circumvent the above problem, a sodium hydroxide solution of approximate molarity is prepared first. This solution is titrated with an acid whose molarity is known with greater accuracy. The data from this titration is then used to calculate a more accurate value for the molarity of the sodium hydroxide solution.

The acid that will be used to determine the molarity of the sodium hydroxide is referred to as the **Primary Standard**. The primary standard must be chosen carefully and it must be a chemical that can be weighed accurately. A commonly used primary standard for titration with sodium hydroxide solution is the weak acid potassium hydrogen phthalate or KHP ($C_8H_5O_4K$).

The reaction between KHP and NaOH is given below:

$C_8H_5O_4K + NaOH \rightarrow C_8H_4O_4KNa + H_2O$

In this reaction as well, one mole of KHP completely reacts with one mole of NaOH. The titration of NaOH with KHP involves adding NaOH from the burette to a known volume of KHP. The molarity of the KHP solution is determined from the mass and volume of KHP used to prepare the KHP solution. The data from the titration is then used to calculate the molarity of the NaOH.

Preparation of primary standard solution

The moles of acetic acid in vinegar will be obtained by titrating the acetic acid with a solution of sodium hydroxide of known molarity. The molarity of the sodium hydroxide solution will be determined by titrating it with a solution of KHP of known molarity. Therefore it should be apparent that the accuracy of the results of the experiment depends on the accuracy of the molarity of the KHP. The KHP solution should be prepared using a volumetric flask. When using a volumetric flask, care must be taken to avoid crossing the calibrated mark.

Experimental Design

A solution of sodium hydroxide whose concentration is known approximately will be provided for this experiment. The exact molarity of this sodium hydroxide solution will be determined by titrating it with a solution of KHP that must be prepared by the experimenter. Once the molarity of the sodium hydroxide solution is determined, it will then be used to titrate the acetic acid in the vinegar. Phenolphthalein is used as an indicator in both of these titrations.

Reagents and Supplies

1.0 M aqueous sodium hydroxide, solid potassium hydrogen phthalate, commercial vinegar solution, 1% and 0.25% phenolphthalein solution

25-mL volumetric flask

(See posted Material Safety Data Sheets)

Procedure

PART 1: STANDARDIZATION OF AQUEOUS SODIUM HYDROXIDE SOLUTION

1. Calculate the volume of 1.0 M aqueous sodium hydroxide solution needed to prepare 50-mL of an approximately 0.10 M aqueous sodium hydroxide solution.

2. Measure the volume of 1.0M aqueous sodium hydroxide solution calculated in step 1 using a 10-mL graduated cylinder. Pour this volume into a 125-mL Erlenmyer flask and dilute the solution with enough deionized water to a total volume of 50-mL.

3. Label the flask with your name, date, and 0.1M aqueous sodium hydroxide (do not use chemical symbols). Labels can be found in the black utility kit. At the end of the period save this solution by stoppering the flask with a #5 stopper obtained from the stockroom and Parafilm (also found in the black utility kit). The solution should be stored in secondary containment as directed by your instructor.

4. Calculate the mass of KHP needed to prepare 25.00 mL of 0.100 M KHP.

5. Weigh an amount of KHP as close as possible to the calculated mass in step 2 and record the exact mass of KHP measured.

6. Transfer the solid KHP into a 25-mL volumetric flask. Note the number of significant figures used for the volume of the 25-mL volumetric flask.

7. Add a small amount of deionized water into the 25-mL volumetric flask containing the solid KHP and swirl the flask until all the KHP is completely dissolved.

8. Add one drop of **1%-phenolphthalein** solution into the volumetric flask.

9. Add deionized water up to the 25-mL mark of the volumetric flask, cover the flask and mix the contents carefully (avoid spills).

10. Clamp two microburettes to a burette stand and label one burette as “NaOH” and the other burette as “KHP”.

11. Condition and fill each burette with the respective reagent.

12. Record the initial burette readings of both the burettes.

13. Dispense a little over 1-mL of KHP solution into a small Erlenmeyer flask and record the exact final burette reading.

14. Rinse the sides of the Erlenmeyer flask with deionized water from a squirt bottle.

15. Titrate the KHP solution in the Erlenmeyer flask with the NaOH solution until a permanent pale pink color is obtained. After the addition of each drop of NaOH, be sure to swirl the Erlenmeyer flask thoroughly to ensure mixing of the reagents. In case any NaOH solution falls on the side of the Erlenmeyer flask, rinse the sides of the flask with deionized water.

16. Repeat steps 10-13 at least two more times.

PART 2: TITRATION OF THE ACETIC ACID IN VINEGAR WITH THE STANDARDIZED SODIUM HYDROXIDE SOLUTION

1. Use the same NaOH solution as in part 1 of this experiment.

2. Obtain approximately 10-mL of commercial vinegar solution.

3. Clamp two microburettes to a burette stand and label one burette as “NaOH” and the other burette as “vinegar”.

4. Condition each burette with the respective reagent.

5. Record the initial burette readings of both the burettes.

6. Obtain a clean, dry, small Erlenmeyer flask.

7. Dispense approximately 0.1-mL of vinegar into the Erlenmeyer flask.

8. Record the mass of the Erlenmeyer flask with the vinegar.

9. Add one drop of **0.25%-phenolphthalein** solution into the Erlenmeyer flask containing the vinegar.

10. Rinse the sides of the Erlenmeyer flask with deionized water.

11. Titrate the contents of the Erlenmeyer flask with the NaOH solution until a permanent pale pink color is obtained (Refer to step 13 of part 1).

12. Record the final burette reading of the NaOH burette.

13. Repeat steps 5-13 two or three more times.

14. Dispose all waste into appropriate waste disposal containers as instructed by your instructor.

Data Table

PART 1: STANDARDIZATION OF AQUEOUS SODIUM HYDROXIDE SOLUTION

Mass of KHP (grams)	
Volume of KHP solution (mL)	25.00

NaOH solution

	Trial 1	**Trial 2**	**Trial 3**	**Trial 4**
Initial burette reading (mL)				
Final burette reading (mL)				
Volume Used (mL)				

KHP solution

	Trial 1	**Trial 2**	**Trial 3**	**Trial 4**
Initial burette reading (mL)				
Final burette reading (mL)				
Volume Used (mL)				

PART 2: TITRATION OF THE ACETIC ACID IN VINEGAR WITH THE STANDARDIZED SODIUM HYDROXIDE SOLUTION

Vinegar

	Trial 1	**Trial 2**	**Trial 3**	**Trial 4**
Initial burette reading (mL)				
Final burette reading (mL)				
Volume Used (mL)				

NaOH solution

	Trial 1	**Trial 2**	**Trial 3**	**Trial 4**
Initial burette reading (mL)				
Final burette reading (mL)				
Volume Used (mL)				

Calculations

PART 1: STANDARDIZATION OF AQUEOUS SODIUM HYDROXIDE SOLUTION

Molar mass of KHP ($C_8H_5O_4K$) =

Mass of KHP =

Moles of KHP =

Volume of KHP solution prepared = 25.00 mL = 0.02500 L

Molarity of KHP = $\frac{moles}{Volume}$ =

	Trial 1	**Trial 2**	**Trial 3**	**Trial 4**
Volume of KHP (liters)				
Volume of NaOH (liters)				

Molarity of NaOH (show calculation for each trial):
$C_8H_5O_4K + NaOH \rightarrow C_8H_4O_4KNa + H_2O$

Trial 1

Trial 2

Trial 3

Trial 4

	Molarity of NaOH
Trial 1	
Trial 2	
Trial 3	
Trial 4	
Average	

PART 2: TITRATION OF THE ACETIC ACID IN VINEGAR WITH THE STANDARDIZED SODIUM HYDROXIDE SOLUTION

$CH_3COOH_{(aq)} + NaOH_{(aq)} \rightarrow CH_3COONa_{(aq)} + H_2O_{(l)}$

At equivalence point: moles of NaOH = moles of CH_3COOH

Average molarity of NaOH (from part 1) =

	Trial 1	**Trial 2**	**Trial 3**	**Trial 4**
Volume of NaOH (liters)				
Moles of NaOH (M × V)				
Volume of CH_3COOH (liters)				
Moles of CH_3COOH				
Molar Mass of CH_3COOH (g/mol)				
Mass of CH_3COOH (grams)				
Mass of vinegar sample (grams) (d=1.005g/mL)				
Mass percent of acetic acid in vinegar (%)				

Average mass percent of acetic acid in vinegar = _____