Experiment 8

Standardization of a Base, Mass Percent of an Acid

Pre-Lab Assignment

Before coming to lab:

- Read the lab thoroughly.
- Answer the pre-lab questions that appear at the end of this lab exercise.

Purpose

An unknown solution of sodium hydroxide with be standardized to determine its molarity by titration with pure potassium hydrogen phthalate (KHP). The standardized NaOH solution will then be used to determine the mass percent of KHP in an unknown mixture.

Background

The reaction of an acid and a base to form a salt and water is known as neutralization. In this experiment, potassium hydrogen phthalate (KHP) is used as the acid. It has a molecular weight of 204.22 g/mol and is monoprotic, meaning it has one acidic hydrogen per molecule. It reacts with sodium hydroxide (NaOH) by the following balanced equation (Eqn. 1).

$$HKC_8H_4O_4(aq) + NaOH(aq) \rightarrow NaKC_8H_4O_4(aq) + H_2O(l) \qquad \text{Eqn. 1}$$

Titration is the process of measuring the volume required for one reagent to react with a pre-measured volume or weight of another reagent. This technique will be used in two ways. In Part I, the molarity of an unknown NaOH solution will be determined by titrating it with a pre-weighed sample of KHP dissolved in water. In Part II, the mass percent of KHP present in an impure mixture will be determined by titrating it with a NaOH solution of known molarity.

During a titration, one reagent is added in very small amounts to the other until the reaction is just complete. At this point, the reagents are present in stoichiometrically equal amounts and is called **equivalence point**. An indicator, any substance that changes color due to changes in conditions such as pH, is added to one of the reagents. The point at which the indicator in the reaction solution changes color is called the **end point**. This is often close to but not always the same as equivalence point. For this experiment, the indicator used will be phenolphthalein which is colorless in acidic and neutral solution but turns pink in alkaline solution.

Standardizing a solution is the process of calculating its concentration by reacting it with pre-measured amounts of another known compound. It is good practice to run multiple trials and use the average concentration to ensure your results are as precise as possible.

Example Problem: Finding the Concentration of an Unknown Solution

A 1.505 g sample of KHP was titrated with an unstandardized solution of NaOH. The initial buret reading was 0.23 mL and the final was 18.62 mL. Find the concentration of NaOH in M.

Step 1: Find the moles of KHP that reacted

$$1.505 \text{ g KHP} \times \frac{1 \text{ mol KHP}}{204.22 \text{ g KHP}} = 0.007345 \text{ mols KHP}$$

Step 2: Find the moles of NaOH that reacted

$$0.007345 \text{ mols KHP} \times \frac{1 \text{ mol NaOH}}{1 \text{ mol KHP}} = 0.007345 \text{ mols NaOH}$$

Step 3: Find the volume of NaOH used

$$18.62 \text{ mL} - 0.23 \text{ mL} = 18.39 \text{ mL} \times \frac{1 \text{ L}}{1000 \text{ mL}} = 0.01839 \text{ L NaOH}$$

Step 4: Find the molarity of NaOH

$$\frac{0.007345 \text{ mols NaOH}}{0.01839 \text{ L NaOH}} = 0.4007 \text{ M}$$

Once the solution's concentration is known it can then be used to determine the amounts of other unknown reagents.

Example Problem: Finding the Mass Percent of an Unknown Mixture

A 2.650 g mixture of KHP and other unreactive substances was titrated with the same NaOH solution from the previous example. The initial buret reading was 1.24 mL and the final was 20.35 mL. Find the mass percent of KHP in the sample.

Step 1: Find the volume of NaOH that reacted

$$20.35 \text{ mL} - 1.24 \text{ mL} = 19.11 \text{ mL} \times \frac{1 \text{ L}}{1000 \text{ mL}} = 0.01911 \text{ L NaOH}$$

Step 2: Find the moles of NaOH that reacted

$$0.01911 \text{ L NaOH} \times \frac{0.4007 \text{ mols NaOH}}{1 \text{ L NaOH}} = 0.007657 \text{ mols NaOH}$$

Step 3: Find the moles of KHP that reacted

$$0.007657 \text{ mols NaOH} \times \frac{1 \text{ mol KHP}}{1 \text{ mol NaOH}} = 0.007657 \text{ mols KHP}$$

Step 4: Find the grams of KHP that were present

$$0.007657 \text{ mols KHP} \times \frac{204.22 \text{ g KHP}}{1 \text{ mol KHP}} = 1.564 \text{ g KHP}$$

Step 5: Find the mass percent of KHP in the original sample

$$\frac{1.564 \text{ g KHP}}{2.650 \text{ g sample}} \times 100 = 59.01\% \text{ KHP}$$

Procedure

Part I: Standardization of a Base

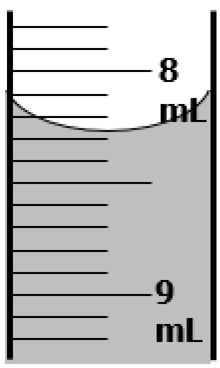
1. Using a weigh boat, measure out between 0.25-0.5 grams of solid KHP. Record accurately the actual weight in your data sheet.

2. Carefully transfer the KHP to a 125 mL Erlenmeyer flask and add about 30 mL of deionized water. If some KHP is sticking to the walls of the flask or the weigh boat, use your wash bottle to spray deionized water on it to wash it down.

3. Swirl the flask to ensure all the KHP dissolves. If some does not dissolve, warm the flask gently.

4. Add 2-3 drops of phenolphthalein solution to the Erlenmeyer flask and mix.

5. Clean a buret thoroughly with some NaOH, then dispose of this solution in the appropriate waste container. Use new NaOH solution to fill it to some relatively convenient position. Ensure that there are no air bubbles in the buret or its tip. Record the initial buret reading to the hundredths place in your data sheet. (Fig. 1)



Note: Do not fill the buret over the 0.00 mL or let it drain past the 50.00 mL mark.

Figure 1: Read the buret from top of the cylinder to the bottom of the meniscus. Volume shown: 8.28 mL

6. Place the Erlenmeyer flask on top of a white piece of paper and slide both underneath the buret.

7. **Slowly** add small amounts of NaOH solution to the Erlenmeyer flask. Swirl after every new portion is added. Continue to add NaOH until the solution in the flask turns fully pink and persists for at least 30 seconds. Record the final buret reading on your data sheet.

Note: the endpoint should be the ***faintest*** *pink possible. Darker colors indicate a larger discrepancy between end point and equivalence point (Fig. 2).*

Figure 2: Three samples show titrations (1) before endpoint (2) at endpoint and (3) past endpoint

8. Dispose of the titrated sample properly. Repeat steps 1-7 for a total of 3 trials.

Note: you do not need to refill your buret to the top for each trial so long as it has enough solution to complete your next titration.

9. Calculate the M of NaOH for each of your three trials. They should agree within 0.01 M. If they do not, repeat trials until you have three that agree. Find the average molarity.

10. Neutralized, titrated samples can be disposed of down the sink while flushed with water. Excess or waste NaOH should be disposed of in labeled waste containers.

Part II: Mass Percent of an Acid

1. Using a weigh boat, measure out 0.5-0.75 g of your solid unknown mixture. Record accurately the actual weight on your data sheet.

2. Repeat Steps 2-8 from Part I.

3. Calculate the mass percent of KHP for each of your three trials. They should agree within 5%. If they do not, repeat trials until you have three that agree. Find the average mass percent of KHP in your solid unknown.

Experiment 8—Data Sheet

Name: ____

Part I: Standardization of a Base **NaOH Solution:** __________________

	Trial One	Trial Two	Trial Three
1. Mass weigh boat (g)	___________	___________	___________
2. Mass weigh boat + KHP (g)	___________	___________	___________
3. Final buret reading (mL)	___________	___________	___________
4. Initial buret reading (mL)	___________	___________	___________
5. Mass KHP (g)	___________	___________	___________

show calculation:

6. Moles KHP (mols) ___________ ___________ ___________

show calculation:

7. Moles NaOH (mols) ___________ ___________ ___________

show calculation:

8. Volume NaOH Used (mL) ___________ ___________ ___________

show calculation:

9. Volume NaOH Used (L) ___________ ___________ ___________

show calculation:

10. Molarity NaOH (M) __________ __________ __________
show calculation:

11. Average Molarity NaOH (M) __________
show calculation:

Part II: Mass Percent of an Acid **KHP Unknown:** _______________

	Trial One	**Trial Two**	**Trial Three**
1. Mass weigh boat (g)	__________	__________	__________
2. Mass weigh boat + sample (g)	__________	__________	__________
3. Final buret reading (mL)	__________	__________	__________
4. Initial buret reading (mL)	__________	__________	__________
5. Volume NaOH Used (mL)	__________	__________	__________

show calculation:

6. Volume NaOH Used (L) __________ __________ __________
show calculation:

7. Moles NaOH (mols) __________ __________ __________
show calculation:

8. Moles KHP (mols) __________ __________ __________
show calculation:

9. Mass KHP (g) ____________ ____________ ____________
show calculation:

10. Mass Sample (g) ____________ ____________ ____________
show calculation:

11. Mass Percent KHP (%) ____________ ____________ ____________
show calculation:

12. Average Mass Percent KHP (%) ____________ ____________ ____________
show calculation:

Experiment 8—Post-Lab Assignment

1. In Part II, if an air bubble is trapped in the buret tip when measuring initial volume but disappears during the titration, will this change the calculated value of the mass percent of your unknown? If so, will it make it larger or smaller than the true value? Explain.

2. Student A titrates all three samples of KHP from Part I to the same dark magenta endpoint and Student B titrations all three samples of KHP from Part I to the same light pink endpoint. Which student (A, B, both, or neither) will have more accurate results for their calculated molarity of NaOH solution? Which student will have more precise results? Explain.

3. In Part I, the weight of dry KHP is measured as accurately as possible; however, the volume of water in which it was dissolved is not recorded. Could you add extra water to the NaOH solution without affecting your data? Explain.

4. A 0.289 g sample of pure KHP is dissolved in 100 mL of deionized water. If 10.0 mL of NaOH solution is required to reach the equivalence point, calculate the molarity of the NaOH solution.

5. The same NaOH solution from Question 4 is then used to titrate 0.567 g of an unknown mixture that contains some amount of KHP and the rest unreactive substances that has been dissolved in 100 mL of deionized water. If 12.3 mL of NaOH solution is required to reach the equivalence point, calculate the mass percent of KHP in the unknown mixture.

Experiment 8—Pre-Lab Assignment

Name: ______________________________________

For all calculations, show all work and draw a box around the final answers.

1. Explain the difference between **end point** and **equivalence point**.

2. Calculate the molar mass of potassium hydrogen phthalate, KHP ($HKC_8H_4O_4$).

3. Write the balanced neutralization reaction occurring in today's experiment.

4. A 0.394 g sample of pure KHP is dissolved in 100 mL of deionized water. If this sample requires 13.9 mL of an unknown solution of NaOH to reach equivalence point, what is the molarity of the NaOH solution?

5. If the solid KHP is dissolved in 45.0 mL of water instead of the recommended 30.0 mL, how does this change (increase, decrease, no effect) the calculated molarity of the NaOH solution? Explain.

6. What is phenolphthalein's color in an acidic solution? ____________________

What is phenolphthalein's color in a neutral solution? ____________________

What is phenolphthalein's color in a basic solution? ____________________

7. A 0.7497 g sample of a mixture of KHP and other unreactive substances requires 23.7 mL of 0.1189 M NaOH solution to reach equivalence point. What is the mass percent of KHP in the mixture?