CHEM 1412 | # Lab – 6 | **Dr. Pahlavan**

Acid – Base Titration
Determination of the Purity of KHP
(Potassium Hydrogen Phthalate)

EQUIPMENT and CHEMICALS

dilute sodium hydroxide, 0.10 M NaOH
potassium hydrogen phthalate (KHP), solid
Phenolphthalein indicator.
unknown KHP
buret clamp (or utility clamp)
small, plastic funnel (optional)
50 mL buret
50 mL graduated cylinder
beakers (100 mL, 150 ml)

Purpose:

a) Determine the percent of potassium acid phthalate (KHP) in an unknown sample by means of an acid base titration with NaOH.
b) To standardize a sodium hydroxide solution with potassium hydrogen phthalate.
c) To determine the molar concentration and mass/mass percent concentration of acetic acid in an unknown vinegar solution.
d) To gain proficiency in the laboratory technique of titration.

INTRODUCTION

In this experiment, a NaOH solution of known concentration will be used to titrate measured masses of a sample containing an unknown amount of potassium hydrogen phthalate (abbreviated KHP but the formula is $K\mathbf{H}C_8H_4O_4$). KHP and the strong base NaOH undergo an acid/base reaction with the following molecular equation:

$$\mathbf{KHC_8H_4O_4 + NaOH \rightarrow H_2O + KNaC_8H_4O_4}$$

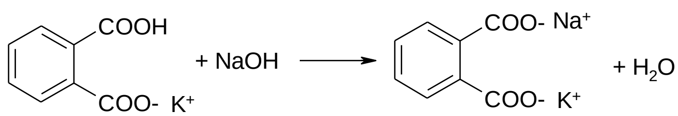
which corresponds to the following net ionic equation?

$$HC_8H_4O_4^- + OH^- \rightarrow H_2O + C_8H_4O_4^{2-}$$

Since there is only one acidic hydrogen in KHP (the one that is shown in boldface in the above formula), an equal number of moles of the strong monoprotic base NaOH and the amphiprotic KHP will react. (Although we are primarily interested in the acidic properties of KHP in this experiment, since KHP is amphiprotic it can also react with acids, in which case a diprotic acid named phthalic acid, $\mathbf{H_2}C_8H_4O_4$ is produced.) The volume of your NaOH solution needed to react with the KHP will be determined by a titration. Phenolphthalein, which changes from colorless to pink when enough NaOH has been added to react with all the KHP present, is used as an indicator for the titration.

The number of moles of sodium hydroxide used in each titration reaction will be determined from known molarity and measured volume of the sodium hydroxide solution. The moles NaOH used in the reaction can be converted to moles of KHP used in the reaction, and then the grams of KHP present in the previously weighed sample can be determined. This mass of KHP along with the mass of sample used in the titration allows calculation of the percent KHP in the unknown.

KHP + NaOH → KNaP + H_2O



Example 1- A 0.905 g sample of KHP (204.23 g/mol) is dissolved in water and titrated with 19.90 mL of NaOH solution to a phenolphthalein endpoint. Find the molarity of the NaOH solution.

Solution: Referring to the preceding equation for the reaction and applying the rules of stoichiometry, we have

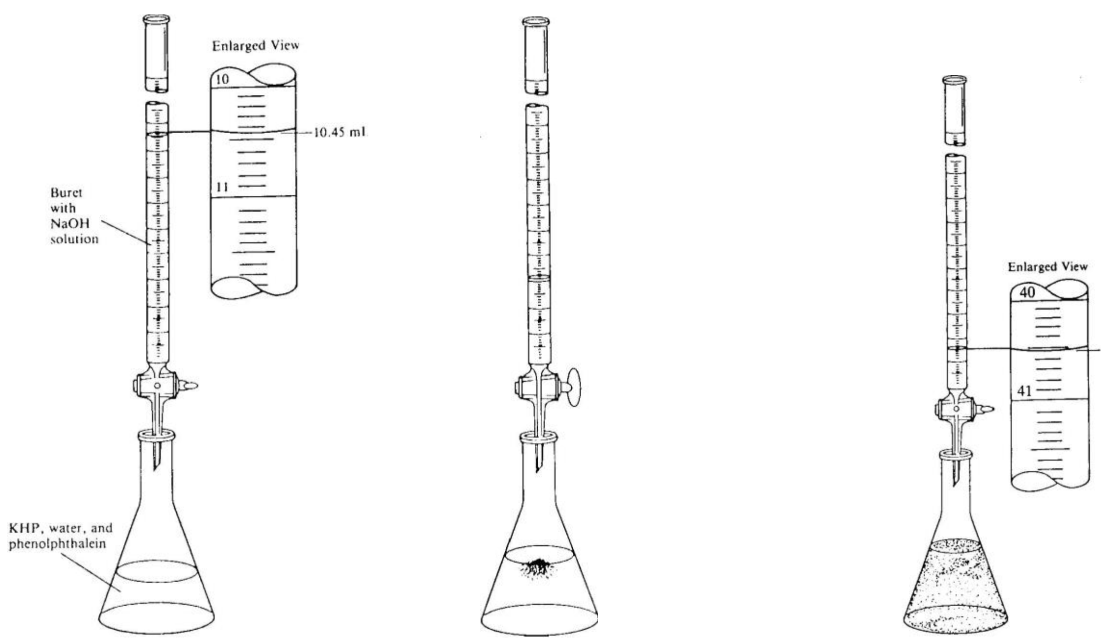
M (NaOH) = (g KHP)(1 mol KHP / 204.23 g) (1 mol NaOH / 1 mol KHP) / (V_L of NaOH)

$$(0.905\text{ g KHP})\left(\frac{1\text{mol KHP}}{204.23\text{ g KHP}}\right)\left(\frac{1\text{mol }\_\_}{1\text{mol KHP}}\right) = 0.00443\text{ mol NaOH}$$

The molarity of the NaOH is found as follows:

$$\frac{0.00443\text{ mol NaOH}}{19.90\text{ ml solution}} \times \frac{1000\text{ ml}}{1\text{ L}} = \frac{0.223\text{ mol NaOH}}{1\text{ L solution}} = \mathbf{0.223\ M\ NaOH}$$

In this example, the concentration of the standard NaOH solution is 0.223 M.



(a) (b) (c)

Figure -1 Apparatus for the Titration of an Acid (KHP) with a Base (NaOH)

(a) Read the initial volume of NaOH in the buret (10.45 mL). (b) A **flash pink** indicates an approaching endpoint. (c) A permanent(light) pink color signals the final endpoint. Read the final volume of NaOH in the buret (40.55 mL). The volume of NaOH used in the titration is: (40.55 mL - 10.45 mL) = 30.10 mL.
After standardizing the sodium hydroxide solution, we will then determine the KHP concentration in an unknown KHP. A sample of KHP will be titrated with the standardized sodium hydroxide to a phenolphthalein endpoint. The equation for the reaction is

$$KHP + NaOH \rightarrow KNaP + H_2O$$

Example 2 - The titration of a 10.0 mL vinegar sample requires 29.05 mL of standard 0.223 M NaOH. Calculate the (a) molarity and (b) mass/mass percent concentration of acetic acid.

Solution: We can calculate the moles of acetic acid from the moles of NaOH solution:

(29.05 ml solution) (0.223 mol NaOH /1000 ml)(1 mol $HC_2H_3O_2$ / 1 mol NaOH) = 0.00648 mol

(a) The molar concentration of $HC_2H_3O_2$ is

(0.00648 mol $HC_2H_3O_2$/10.0 ml solution)(1000 ml/1 L) = 0.64 M

(b) To calculate the m/m % concentration, we must know the density of the vinegar (1.01 g/mL) and the molar mass of acetic acid (60.06 g/mol).

$$\left(\frac{0.648\text{mol } HC_2H_30_2}{1000 \text{ mL solution}}\right) \times \left(\frac{60.06 \text{ g } HC_2H_3O_2}{1 \text{ mol } 1\ HC_2H_3O_2}\right) \times \left(\frac{1 \text{ mL solution}}{1.01 \text{ g solution}}\right) \times 100$$

= **3.85% $HC_2H_30_2$**

Example 3 - Assume we used 25 ml of NaOH to reach the endpoint using 0.1000 mol/L NaOH for titration of unknown sample of KHP. Calculate the percentage KHP in the unknown sample mass of 0.9735 g.

Solution- The number moles of acid the following formula is used:

0.1000 mol/L NaOH x 0.02500 L NaOH x 1 mol KHP/1 mol NaOH = 0.0025 mol KHP

To determine the percent KHP in an unknown sample mass of 0.9735 g, the following calculation is used:

0.0025 mol KHP x 204.23 g KHP/1 mol KHP x 1/0.9735 g unknown x 100% = **52.44% KHP**

Example 4 - Assume 0.5472 g of 100% pure KHP is titrated with a NaOH solution of which the molarity is not known. Calculate the molarity of sodium hydroxide solution if 37.42 mL (0.03742 L) of sodium hydroxide were used to titrate the KHP.

Solution - The following equation is used to calculate the molarity of the NaOH solution. Therefore:

(0.5472 g KHP)(1 mol KHP/204.23 g KHP)(1 mol NaOH/1 mol KHP)(1/0.03742 L NaOH)
= 0.09771 mol NaOH/1 L NaOH = **0.09771 M**

NaOH Preparation- Prepare a ~0.1 M NaOH solution and store it in a polyethylene bottle (not glass). You will standardize this solution, so the preparation need not be exact. Before use, be sure your NaOH solution is fully dissolved, and well mixed by shaking. Rinse your burette with your NaOH solution, and then fill it, being sure to remove any air bubbles.

SAFETY CAUTION: NaOH is strongly basic and can damage skin and clothing. Handle it carefully and clean up any spill immediately. **Note**: Carefully add NaOH solution to the funnel so as to not overfill the buret. Drain some NaOH through the tip of the buret to clear any air bubbles.

Procedure

Part I - Standardization of NaOH

Label three 250 mL Erlenmeyer flasks #1, #2, and #3. Accurately weigh by difference approximately 0.40-0.50 g of KHP (100%; NOT your unknown) into each of the flasks. Add 50 mL of distilled water to each flask and swirl gently (may required heat) as necessary to dissolve the KHP crystals. Add two drops of phenolphthalein indicator to each flask.

Position Erlenmeyer flask #1 under the buret as shown in Figure -1. Record the bottom of the meniscus as the initial buret reading. Titrate the KHP sample to a light pink endpoint. Record the final buret reading. Refill the buret, record the initial buret reading, add a drop of phenolphthalein indicator to flask #2, titrate the KHP sample, and record the final buret reading.

Refill the buret, record the initial buret reading, add a drop of phenolphthalein indicator to flask #3, titrate the KHP sample, and record the final buret reading.

Calculations

1. Use the mass of pure KHP added to each flask, and the volume of sodium hydroxide solution used in each of the three standardization titrations to calculate the molarity (or Normality) of the sodium hydroxide solution for each trial. calculate the concentration of sodium hydroxide used in each titration.

   M (NaOH) = (g KHP)(1 mol KHP / 204.23 g) (1 mol NaOH / 1 mol KHP) / (V_L of NaOH)

2. Calculate the average sodium hydroxide concentration and the standard deviation of the results. Record the average molarity in the report form.

Part II - Percentage purity of KHP

Repeat the experiment with unknown samples. Weigh by difference approximately 0.40 -0.50 g of unknown into each of three separate clean 250 mL Erlenmeyer flasks. Record all masses to 2 decimal places. Repeat the procedure by adding about 75-100 mL deionized water to each flask, and swirl gently to dissolve the sample. Add two drops of phenolphthalein indicator to each flask, and titrate. Record the volume required.

Calculations

1. Use the mass of impure KHP(unknown) added to each flask and the volume of the NaOH solution to calculate the purity of the impure sample of KHP. Again, calculate the standard deviation of the results.
2. Use the average sodium hydroxide concentration, the end point volume for each titration, and the volume of sample.
3. Calculate the average KHP percentage and the standard deviation of the results.
4. Check your calculations for errors. Is your answer reasonable?
   Report on the form provided the values as well as the average concentrations found for the sodium hydroxide and KHP solutions. Include the standard deviations and the relative standard deviations for both standardizations.

CHEM 1412 **Lab – 6** **Dr. Pahlavan**

Determination of the Purity of KHP

REPORT FORM

Name ______________________________

Instructor __________________________

Date ____________

Part I. NaOH Standardization

mass of Erlenmeyer flask	_______g	________g	________g
flask + KHP	_______g	________g	________g
mass of KHP	_______g	________g	________g
initial buret reading	_______ml	________ml	________ml
final buret reading	_______ml	________ml	________ml
volume NaOH	_______ml	________ml	________ml
Molarity of NaOH	_______M	_______M	__________M
Average molarity of NaOH	_______M		

Calculation

Trial #	mass KHP (g)	volume NaOH (mL)	molarity (M)

Average Molarity: _______________ Standard Deviation: ________________

Part II. KHP Percentage determination (Unknown # ______)

mass of Erlenmeyer flask	_______g	________g	________g
flask + unknown sample	_______g	________g	________g
mass of unknown sample	_______g	________g	________g
initial buret reading	_______ml	________ml	________ml
final buret reading	______ml	________ml	________ml
volume NaOH	_______ml	________ml	________ml
gr Weight KHP	_______g	________g	________g
% KHP	_______%	________%	________%

Average % KHP ______________________

Calculation

Trial #	sample mass (g)	volume NaOH (mL)	mass KHP (g)	wt % KHP

Average Weight %: ____________ Standard Deviation: _____________

CHEM 1412 # Lab – 6 **Dr. Pahlavan**

Pre Laboratory Review Questions and Exercises

Due **before lab** begins. Answer on a separate sheet of paper.

Name ________________________

Date_________________________

1. What is the difference between a primary and a secondary standard? Give an example of each.

2. Why must air bubbles be expelled from the buret tip?

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3. 0.3043 g of pure KHP was weighed out and titrated to an end point with 15.12 mL of a NaOH solution that was approximately 0.1 **M**. What is the exactly concentration of the NaOH titrant?

4. 0.5366 g of an KHP sample of unknown purity was massed. The sample was dissolved in approximately 100 mL of distilled, degassed water and indicator was added. The end point was reached after 21.35 mL of 0.09854 **M** NaOH solution was titrated into the solution. What is the percentage of KHP in the original sample?

5. What is the molarity of a NaOH solution if 32.47 mL is required to titrate 0.6013 g of potassium hydrogen phthalate ($C_8H_5O_4K$)?

CHEM 1412 # Lab – 6 **Dr. Pahlavan**

Post- laboratory Questions and Exercises
Due after completing lab. Answer in the space provided.

Name_________________________

Date _________________________

1. Write the formula unit and the net-ionic equations to describe the reaction between potassium hydrogen phthalate (KHP) and cesium hydroxide. Use $C_8H_5O_4K$ as the formula for potassium hydrogen phthalate, $C_8H_5O_4^-$ to represent the hydrogen phthalate ion and so forth.

2. A student needs to determine by titration with NaOH the precise % KHP in an unknown sample that is thought to contain approximately 50% KHP. Appoximately what mass of sample should the student use in order to use about 20 mL of 0.1005 M NaOH to reach the endpoint of the titration?

3. If the student in problem 2 actually used 0.847 g sample of the impure KHP and the endpoint was reached after 19.82 mL of the sodium hydroxide solution was added, what is the percent KHP in the unknown sample to four significant figures (Yes, we are abandoning rules of thumb for significant figures for this result)?

4. A 0.5123-g KHP (M.W. 204.23) sample was dissolved in about 25 mL of distilled water, and titrated to the phenolphthalein end point with 28.75 mL of a sodium hydroxide solution. Calculate the molar concentration of the hydroxide solution.

5. 19.80 mL NaOH is required to titrate 0.6013 g of potassium hydrogen phthalate ($C_8H_5O_4K$). How many ml NaOH required to titrate 25.0 ml 0.10 M HCl solution?