<u>EXPERIMENT 12 A: STANDARDIZATION OF A SODIUM HYDROXIDE SOLUTION</u>
OBJECTIVE:
   Sodium hydroxide solution of about 0.2 M is prepared in order to be used in Exp 12B. The solution is then standardized, that is, its concentration is accurately determined, by titrating it against a weighed sample of known acidic salt, potassium acid phthalate.

INTRODUCTION:
Sodium hydroxide is deliquescent (absorbs moisture from the atmosphere) solid. It cannot be weighed accurately. Therefore, it is not possible to prepare a standard solution of sodium hydroxide of accurately known concentration by weighing NaOH. A sodium hydroxide solution of approximate concentration (0.2 M) is to be prepared. It is then standardized by titrating it against an accurately weighed sample of potassium acid phthalate (KHP), $HOOC\text{-}C_6H_4\text{-}COOK$, which is a primary standard acidic substance. A primary standard substance can be weighed accurately because it is stable (does not decompose) and not hygroscopic (does not absorb moisture from the atmosphere). The reaction between NaOH and KHP (molar mass 204.23 g/mole) is as follows:

$$NaOH + HOOC\text{-}C_6H_4\text{-}COOK \rightarrow NaOOC\text{-}C_6H_4\text{-}COOK + H_2O$$
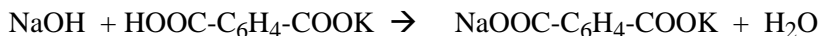
By measuring the volume of the ~0.2M NaOH solution dispensed from the buret that is necessary to react completely with a weighed sample of KHP, the exact concentration of NaOH solution is calculated.

PROCEDURE:
Check out a buret from the stockroom.
A. <u>PREPARATION OF THREE SAMPLES OF KHP</u>
        1) Clean three 125 ml Erlenmeyer flasks and rinse with distilled water. Label the Erlenmeyer flasks as 1, 2, and 3 using a pencil.
        2)   Accurately weigh out by difference three samples of KHP into the three labeled flasks. Each sample should weigh between 0.3g and 0.4 g.

B. <u>PREPARING SODIUM HYDROXIDE SOLUTION OF APPROXIMATE CONCENTRATION</u>
        1. Clean your 250 ml Erlenmeyer flask and rinse it with distilled water.
        2. Meaure between 8 and 8.5 ml of 6M NaOH using a clean, but not necessarily dry 10 ml graduate cylinder. The 6M NaOH reagent bottle is on your bench.
        3. Pour the NaOH solution into the 250 ml Erlenmeyer flask and then add distilled water until the top of the solution is at the flask's neck. Mix the solution well by pouring into a clean but not necessarily dry 400 ml beaker and then pouring it back into the 250 ml Erlenmeyer flask. Do this several times to make certain the solution is thoroughly mixed.
        4. Label the 250 ml Erlenmeyer flask and stopper it with a #6.5 rubber stopper which you will find on the Chem 111 reagent shelf. Do not allow the rubber stopper to come in contact with the NaOH solution. Be sure to keep the solution stoppered when you are not using it (H_2O reacts with CO_2 in the atmosphere forming H_2CO_3). Also, mix the solution well each time before you use it.
<u>NOTE: YOU WILL BE USING THIS SOLUTION IN BOTH, THIS EXPERIMENT AND EXP 12B. IF YOU WASTE THE SOLUTION YOU MAY RUN OUT BEFORE YOU HAVE FINISHED EXP 12B</u>.

C. <u>PREPARATION OF THE BURET</u>

Check out a 25 ml buret from the stockroom.

1. Rinse the buret with tap water, then rinse it with distilled water. Make the final rinsing of the buret with about 3 ml of the NaOH solution you just prepared and make certain that you drain it through the stopcock.

2. Attach the buret with stopcock closed to a buret clamp mounted on a ring stand. Fill the buret above the zero mark with the NaOH solution. Open the stopcock to allow the excess solution to drain. This will aid in removing the air pocket from the tip of the stopcock. Make sure that the tip of the buret is filled with solution and that there are no air bubbles.

D. <u>STANDARDIZATION OF THE NaOH SOLUTION</u>

1. At your bench add <u>*about*</u> 50 ml of distilled water to KHP sample #1. Add 2 drops of phenolphthalein indicator. Swirl to dissolve the KHP completely.

2. On the report sheet, record the initial reading of the NaOH solution in the buret to the nearest 0.02 ml. Ask your instructor to check your reading and initial your report.

3. Place a white sheet of paper under Erlenmeyer flask #1 to facilitate the detection of the end point when noting the color change of the indicator. Titrate KHP acid solution sample #1 by slowly adding NaOH from the buret and constantly swirling the flask until you have reached the end point. This is indicated by the appearance of a faint pink color of phenolphthalein that persists for 30 seconds or more after swirling the flask. Take the final buret reading to the nearest 0.02 ml and record it on your report sheet.

4. Refill the buret. Repeat the titration for the remaining two KHP samples. Record your data on your report sheet.

5. ***STOPPER AND SAVE YOUR NaOH SOLUTION FOR USE IN EXPERIMENT 12B !!!!!***

E. <u>CALCULATION</u>
1. Calculate the molar concentration of the sodium hydroxide solution using your data for each run.

2. Calculate the average molar concentration of the sodium hydroxide solution. ***THIS IS THE VALUE THAT YOU WILL USE IN EXPERIMENT 12B.***

3. Calculate the precision of your experiment as ***standard deviation*** and as ***% deviation***.

Note: If your percent deviation is greater than 2% you will need to carry out additional titrations until you have a minimum of three values for the molar concentration of NaOH that deviate by less than 2%.

EXPERIMENT: Standardization of NaOH   Last   First

                                    Instructor's initial_____

DATA

A) KHP Samples

	1	**2**	**3**
Mass of KHP	g	g	g

Data Approval _____

B) NaOH solution

	1	**2**	**3**
Initial Buret Reading	ml verification_____	ml	ml
Final Buret Reading	ml	ml	ml
Calculated Volume of Base	ml	ml	ml

Data Approval _____

CALCULATIONS:

A) Molarity of NaOH

Sample #1:

Setup:

_____M

Sample #2:

Setup:

_____M

Sample #3:

Setup:

_____M

B) Average molar concentration of NaOH solution.

Setup:

_____M

C) Precision:

Standard deviation:

Setup:

$\pm$ _____ M

Percent deviation:

Setup

_____%

USE THIS PAGE ONLY IF YOU NEED TO DO MORE THAN THREE TITRATIONS

DATA
A) KHP Samples

	4	**5**	**6**
Mass of KHP	g	g	g

Data Approval _____

B) NaoH solution

	4	**5**	**6**
Initial Buret Reading	ml verification_____	ml	ml
Final Buret Reading	ml	ml	ml
Calculated Volume of Base	ml	ml	ml

Data Approval _____

CALCULATIONS:
A) Molarity of NaOH
Sample #4:
Setup:

_____M

Sample #5:
Setup:

_____M

Sample #6:
Setup:

_____M

B) Average molar concentration of NaOH solution.
Setup:

_____M

C) Precision:
Standard deviation:
Setup:

$\pm$ _____ M

Percent deviation:
Setup

_____%

QUESTIONS
1. Why is it important to keep the NaOH solution stoppered at all times when it is not in use?

2. Why is it all right to use a wet flask for the titration?

3. Define:
       a. A primary standard substance

       b. A standard solution

4. $H_2C_2O_4.2H_2O(s)$ is a primary standard substance. 2.3688 g of oxalic acid dihydrate were completely neutralized by 42.56 ml of NaOH solution. Calculate the molar concentration of the NaOH solution. Hint: You must write a balanced equation for the reaction.
Setup:

Answer_____