**Standardization of NaOH with KHP**

**Standardization of NaOH**

Determining the concentration of an acid or base is common lab procedure. A convenient technique for doing this is called titration. This is the process of carefully adding one reactant to another, and measuring how much is needed for their reaction to be complete. In this experiment, a base will be added to an acid until the neutralization reaction is complete. (H+ + OH- → H2O) The point at which the acid has been exactly neutralized by the base is called the *equivalence* point. Determination of the equivalence point is accomplished by using an appropriate indicator, which is a compound that changes color at a hydrogen ion concentration (pH) as close as possible to that present at the equivalence point. This color change is the *endpoint* of the titration. The difference between the endpoint & equivalence point of a titration is usually minimal.

The equivalence point does not necessarily occur at pH 7, which most students tend to think of when they hear “neutralization.” Salts that form can affect the pH. In this experiment, a weak acid will be titrated with a strong base (NaOH), so the equivalence point will be slightly basic. The indicator used will be phenolphthalein, which changes from colorless to pink in the pH range of 8.0-9.8, making it a good indicator to use here.

The objective of this experiment is to standardize (find the exact concentration of) a NaOH solution. Concentration will be expressed in M (moles/L). A solution of NaOH will be prepared and then its exact molarity determined by titration. This is necessary because the exact weight of solid NaOH cannot be determined simply by using the balance. Solid NaOH is hygroscopic, meaning it readily absorbs moisture from the air. Once it has a little moisture it also absorbs carbon dioxide which is always present in air. 2 NaOH(s) + CO2(g) → Na2CO3(aq) + H2O(l)

Therefore solid reagent grade sodium hydroxide is not pure enough to weigh directly. Furthermore, the carbonate ion interferes in acid-base titrations because it is a base, and it tends to make the color change at the end point less sharp. This reaction also takes place in the aqueous phase, where sodium hydroxide in solution is converted to sodium carbonate. This can change the concentration of the standard solution if steps are not taken to minimize the carbon dioxide uptake. It is therefore necessary to prepare sodium hydroxide solutions in such a way that they are free of carbonate impurity. The most convenient method takes advantage of the fact that sodium carbonate is insoluble in

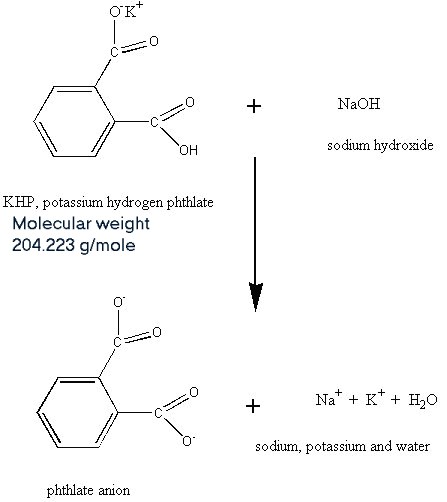
50% NaOH solution. Carbonate free solutions can be obtained simply by diluting 50% NaOH to the required molarity. The solutions are prepared by using CO2 free deionized water. This water is made by boiling the DI water for several minutes to expel the CO2.

You will be provided with a NaOH solution that is approximately 0.5M. It is therefore necessary to measure the concentration of a diluted solution by using it to titrate a known amount of acid. This is called *standardization* of the solution. This characteristic makes NaOH a *secondary* standard, one whose concentration must be determined by comparing it to the concentration of a *primary* standard. A primary standard is a substance of high purity that is stable in the air. When using a primary standard, knowing the mass used, its formula mass, and the volume of solution in which it is dissolved is all that is needed to calculate its exact concentration. Potassium hydrogen phthalate, KHC8H4O4, is the primary standard acid we will use to standardize the NaOH. Potassium hydrogen phthalate is commonly called KHP. KHP is a monoprotic organic acid (contains –COOH carboxyl group).

The chemical reaction between KHP & NaOH is: KHC8H4O4 + NaOH → KNaC8H4O4 + H2O

Notice that one mole of KHP reacts with one mole of NaOH.

See the reaction on the next page using the structural diagram of KHP.

**Procedure:**

A known mass of solid KHP is dissolved in water, phenolphthalein is added, and the NaOH is titrated into the KHP solution until it just turns a light pink. Near the endpoint the solution will begin to show the pink color and it will disappear as the flask is swirled. The light pink color must remain for at least 30 seconds to confirm the endpoint has been reached. This will occur with the addition of one additional drop of NaOH solution. A dark pink/fuchsia color indicates that you have gone beyond the endpoint.

**Part A: NaOH Standardization**

1. Rinse the buret at least twice with some distilled water. Allow it to completely drain into the sink. Rinse it once with 5 - 10 mL of the NaOH solution. This will remove any water remaining in the buret. Fill the buret with the NaOH so it is above the 0.00 mark. Drain the buret until the liquid level is resting one or below the same mark. This ensures that no air bubbles are in the buret tip. Record the initial volume to the nearest 0.00 mL
2. Clean and thoroughly rinse a 125mL Erlenmeyer flask – then rinse it with some DI water from the wash bottle. Put a weigh boat on your balance and tare the balance. Add KHP **carefully** to the weigh boat until you have about 1.5g. Record the exact mass to the nearest **0.001g**. Carefully add the KHP to a 125 mL flask – rinse the KHP from the plastic weigh boat into the flask using your DI wash bottle.

(***Take care not to lose any KHP during the process!***)

1. Dissolve the KHP in about 50-100 mL of DI water (*it does not have to dissolve completely*). This does not change the moles of acid, but just puts them in solution & spreads them out to be neutralized more quickly.

Add 3 drops of phenolphthalein indicator.

1. Lower the buret tip so it is just inside the opening of the flask. Place a white paper under the flask so the endpoint is more visible.
2. Record the initial buret reading for NaOH. Begin titrating by adding NaOH from the buret to the flask. You can add base in 1-2 mL amounts at first, then drop by drop as the pink color starts to linger. The endpoint is reached when one drop of NaOH turns the solution light pink and persists for at least 30 seconds. Record the final buret reading of NaOH. Repeat two more times for a total of three trials. If you miss the endpoint in one of your trials, record the data anyway. Calculating the average will minimize the error.

It is not necessary to refill the buret to zero each trial; just make sure you have enough solution in the buret to complete the trial. (The final volume of trial 1 can be the initial volume of trial 2.)

6) Calculate the molarity of the standardized NaOH solution before starting part B.

**Part B: Analysis of an Impure Sample of KHP**

1) Obtain one of the impure samples of KHP. Open the container and break up any lumps with a stirring rod.

Close the container and shake well to mix. The sample must be uniform! ***(Record the number of the sample.)***

2) Make sure the 125mL flask has been cleaned and rinsed,

3) Weigh about 1.5g of the impure KHP in the weigh boat and transfer it to the flask as before with 50-100mL

of DI water.

4) Titrate the impure sample with the standardized NaOH solution. (The impure sample will contain between

60-85% KHP so less volume of NaOH will be needed to neutralize the sample.)

5) Repeat the titration for a second/third trial.



Reading a Buret: Liquids form a curved meniscus when placed in the buret. The most accurate readings are

obtained by observing the position of the lowest point on

the meniscus. To avoid parallax error, your eye must be

on level with the meniscus. Line up the dark paper strip

so that the front and back edges are in line with the lowest part of the meniscus and take the reading by estimating to the 0.01 mL.



Add 1-2mL at a time until the pink color starts to persist, then add drop by drop until the pink color remains for at least 30 seconds.

Name \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Partner\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Date \_\_\_\_\_\_\_\_

**Standardization of NaOH with KHP**

**Part A: Standardization of NaOH solution** Trial 1 Trial 2 Trial 3

Mass of KHP \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

Final buret volume \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

Initial buret volume \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

mL of NaOH used \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

Molarity of NaOH \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

Average Molarity :\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Show the calculation for the molarity of the NaOH solution for the best trial and for the overall average molarity of the standardized solution. (Molarities should agree within 1%)

**Part B: Analysis of an Impure Sample of KHP** Trial 1 Trial 2 Trial 3

Mass of impure KHP \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

Final buret volume \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

Initial buret volume \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

mL of NaOH used \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

Molarity of NaOH \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

Mass of KHP in sample \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

%KHP in the sample \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_ \_\_\_\_\_\_\_\_\_\_\_

Show the calculation for the mass of KHP in the sample for the best trial. *Unknown # \_\_\_*

Show the calculation for the % KHP in the sample. Calculate your percent error. *Known % \_\_\_\_\_*

