

**Chemistry 123**  
**VOLUMETRIC ANALYSIS**  
**DETERMINATION OF A WEAK ACID**

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**LEARNING OBJECTIVES**

The objectives of this experiment are to...

- prepare a solution containing approximately 0.050 M NaOH.
- standardize the NaOH solution to determine its exact molarity.
- use the standardized NaOH to determine the percentage of a weak acid in an unknown sample.

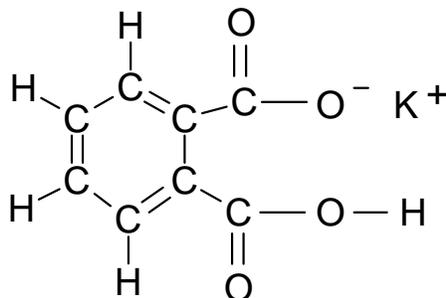
**BACKGROUND**

Quantitative analysis based on volume measurement is known as volumetric analysis. A standard solution – one whose molar concentration is accurately known – is reacted with an unknown sample. From the concentration of the standard solution and the volume required for complete reaction, the amount of the substance to be analyzed can be calculated.

This experiment involves acid-base volumetric analysis. The unknown sample contains a weak acid mixed with a non-acidic, inert material. You are to analyze the sample and determine to four significant figures the percentage of the weak acid originally present in the sample. There are three parts to the analysis.

- (1) An approximately 0.050 M NaOH solution is prepared.
- (2) The NaOH solution is standardized by titrating samples of a pure (primary standard) weak acid with the NaOH solution.
- (3) The standardized NaOH solution is used to titrate unknown samples to determine the percentage of acid present.

The pure acid that will be titrated to standardize your NaOH solution is potassium hydrogen phthalate,  $\text{KC}_8\text{H}_5\text{O}_4$ , (FW: 204.2 g/mol). It has the structure shown below.



For convenience, the formula of potassium hydrogen phthalate is represented as KHP where K is the potassium cation, H the acidic hydrogen in the anion, and P the rest of the anion. (Note: P is *not* phosphorous in this formula.) When dissolved in water, KHP ionizes completely to  $K^+$  and  $HP^-$ . The  $HP^-$  anion is a weak acid and ionizes slightly.



When titrated with NaOH, the  $OH^-$  reacts with  $H^+$  to form water. This disturbs the equilibrium and  $HP^-$  ionizes further to re-establish the equilibrium. Continued addition of NaOH will ultimately lead to complete reaction of all of the  $HP^-$ . The net ionic equation is,



The equivalence point of the titration is detected using the indicator phenolphthalein. Acid-base indicators are weak organic acids or bases whose acid form has a different color than the base form. The form which predominates in solution depends on the pH, so the selection of indicator depends on the pH at the equivalence point. In this titration reaction, the acid form of phenolphthalein (colorless) shifts to the base form (pink) just beyond the equivalence point. The amount of excess NaOH needed to give the color change is very small and will not affect the accuracy of the titration.

The chemistry involved in titrations of your unknown sample with NaOH is identical to that just described, since the unknowns consist of pure KHP mixed with a non-acidic, inert material.

### SAFETY PRECAUTIONS

Wear departmentally approved eye protection at all times in the laboratory. Follow all additional laboratory rules and regulations provided by your instructor. Know the location and proper use of all laboratory safety equipment (safety showers, eye washers, fire extinguishers, etc.). Dispose of all chemicals in the proper waste containers located in the Waste Hood.

A material safety data sheet (MSDS) for each chemical used in this experiment is located in a binder in the lab. You should be familiar with the hazards associated with each chemical, as well as the instructions on safe handling and appropriate disposal. Your instructor will be available to assist you in interpreting this information.

### EXPERIMENTAL PROCEDURE – this lab is to be done individually

#### Drying Standard and Unknown KHP Samples

You will be issued a solid sample of unknown which contains the weak acid, KHP. Record your unknown number in your lab notebook. Add about 2 g of the primary standard KHP to a weighing bottle. Place the unknown vial, cork, weighing bottle and its lid inside a beaker and cover with a watch glass. (The cork and lid should both be off.) Your lab instructor will direct you to a 110°C oven to dry your sample for approximately 45 minutes. Cool the samples for 5 minutes on the bench top and 25 minutes in the desiccator. The lids should be on when storing in a desiccator.

## Preparation of Approximately 0.050 M NaOH

Fill a clean, well-rinsed 1-L plastic bottle with DI H<sub>2</sub>O, leaving about a half inch of air space at the top and invert the bottle several times to make sure that it does not leak. The lab assistant will dispense 3 mL of carbonate-free 50% NaOH solution into the bottle. Cap the bottle and thoroughly mix by repeated inversion of the bottle for at least five minutes. It is extremely important the solution be homogeneous, and the 50% NaOH is very viscous and requires considerable mixing. The resulting solution should be approximately 0.050 M in NaOH.

## Standardizing the NaOH Solution

After the KHP has cooled, accurately weigh (to the nearest 0.0001 g) by difference portions of KHP into each of three 250-mL Erlenmeyer flasks. Each sample should be between 0.35 and 0.45 g. *Directly before beginning titrations*, add to each portion of KHP about 50 mL of DI water and swirl until the solid has dissolved.

Clean your buret thoroughly and rinse several times with the NaOH solution. Make sure the stopcock nut is tight enough to prevent leaking. Fill the buret and drain out enough solution to ensure that no air bubbles are trapped between the stopcock and the tip. When you are sure your buret is working properly, adjust to volume to just below the 0 mL mark.

After taking the initial buret reading, add 4-5 drops of 0.1% phenolphthalein indicator solution to one of the flasks containing dissolved KHP, position the buret so that its tip is 2-3 centimeters inside the flask opening, and titrate with NaOH solution to a stable end-point. You should aim for the faintest pink color which remains after a few seconds of swirling. You can estimate how close you are to the end-point by how long the transient pink color remains in the swirled solution. As you get close, add NaOH drop-by-drop. At the end-point a single drop will change the solution from colorless to a faint pink. Placing a white piece of paper under the flask helps in seeing the color. The color may fade after a while due to absorption of atmospheric CO<sub>2</sub>. Be careful not to expose the NaOH solution to the air unduly while filling the buret, and do not leave it in the buret longer than necessary. Upon reaching the endpoint, determine the volume of NaOH used in the titration to the nearest  $\pm 0.01$  mL, and record the volume.

After the first titration, calculate the approximate volumes of titrant expected for the remaining samples. (Titrant volume and sample mass are proportional.) If you know the approximate titrant volume for the remaining samples, you can add NaOH quickly to within 2-3 mL of the end point, and then approach the end point more cautiously; this saves considerable time.

Calculate the molarity of your NaOH solution to four significant figures for each titration. Also determine the mean value of the molarity, the standard deviation, and the relative standard deviation in parts per thousand. You will be graded on the rsd of your titrations.

## Titration of an Unknown Weak Acid

In determining the best sample size to use for the unknown titrations, several factors need to be kept in mind. First, the *relative* error in a measurement (buret volume measurement or sample weight) is, in general, less if the measurement is large; this would recommend a large sample size. If, however, the sample size is so large that more than one buret full (over 50 mL) of NaOH is required, buret refilling and additional readings will be required which may increase error, so you don't want your sample mass to be too big. But what is *too big*?

Since you don't know the percentage of KHP in your unknown, you will have to estimate the best sample size by making a few reasonable assumptions. Assume your NaOH solution is 0.050 M, (this should be close); assume the sample is 55% KHP (this is less certain); assume the volume of NaOH required is 35 mL (ideally, this volume should be just under 50 mL, but assuming 35 mL allows for some leeway in the assumed % KHP). Now calculate the sample size of the unknown KHP that would be consistent with these assumptions. Keep in mind that this is an approximate calculation; no more than 2 significant figures should be retained, and the range of sample masses may be  $\pm 0.1$  g of your approximate calculation. If you are willing to accept a bit more risk, sample masses within  $\pm 0.2$  g may be satisfactory. The final weighings should still be done to an accuracy of 0.0001g.

Weigh *by difference* three portions of your unknown into 250-mL Erlenmeyer flasks. Dissolve the accurately weighed samples in DI water, add the phenolphthalein indicator, and titrate in exactly the same manner as described above for standardization of the NaOH solution.

Calculate the percentage KHP in each of the three titrated samples to four significant figures. Also determine the mean percentage KHP, the standard deviation, and the relative standard deviation in parts per thousand. You will be graded on the rsd of your titrations as well as the accuracy of your mean percentage KHP compared with the real percentage KHP in the unknown.

**NOTE:** Save your standardized NaOH solution for use in the next experiment.

### Recommended strategy

Two periods are allotted for this experiment. It is recommended that during the first period you do the following:

- (1) Dry the samples of pure KHP and the KHP unknown.
- (2) Weigh the three samples of pure KHP into clean, dry flasks; cover with parafilm for storage between lab periods. (Do not add water before storing!!!)
- (3) Prepare your NaOH solution.
- (4) Perform a practice NaOH standardization titration.

Doing a practice titration will give you added experience in reading the buret accurately and provide familiarity with the endpoint color change. To do a practice titration, there is no need to weigh the KHP accurately. Roughly 0.4 g of KHP weighed on the top-loading balance, added to a 250-mL beaker, and titrated as described above should suffice for the practice titration. **Note:** You do not need to use your dried KHP for this practice titration.

If you have time, you can standardize your NaOH solution, but keep in mind that to yield reliable results, all three standardization titrations must be carried out during the same lab period.

During the second period allotted for the experiment, the three standards and three unknowns should be titrated as described above.

### Laboratory Report

When performing calculations and completing your Summary Sheet, be careful to express all calculated results to proper significant figures and include units. Remember that buret readings should be estimated to 0.01 mL.