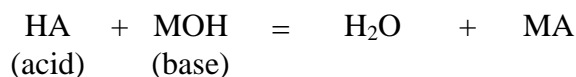


Titration of the Weak Acid Potassium Hydrogen Phthalate (KHP)

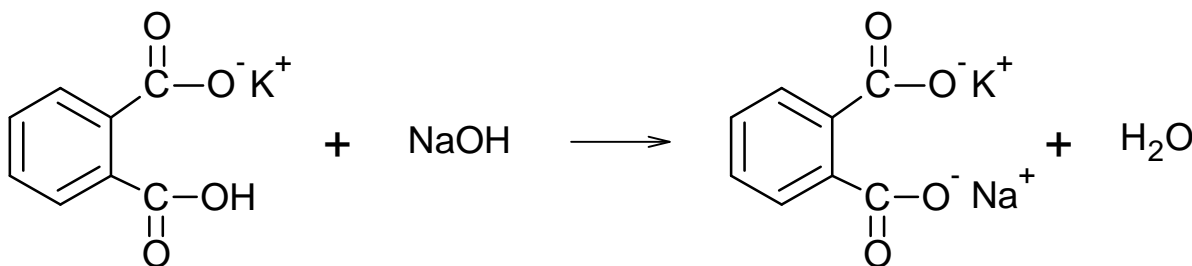
INTRODUCTION

Materials generally considered to possess acidic and/or basic properties are widely distributed in nature and range from simple inorganic materials through organic and biological molecules of great complexity. Since acid-base equilibrium is a general phenomenon, it is advantageous to use it as an analytical tool. Acid-base titrations are conducted by adding a known amount of one reagent (either acid or base) to a sample of the opposite nature (either base or acid) until all available ionizable hydrogen ions of one solution have reacted with all available hydroxide ions in the other to form water. This experiment is designed both to demonstrate the techniques of titrimetric analysis and to explore the relationships involving stoichiometry, pH, and acid-base equilibria.

The general reaction involved in neutralization titrations can be depicted as follows:



In this experiment you will determine the amount of acid present by titration with the strong base NaOH. Since it is hard to prepare a NaOH solution of accurately known concentration directly from the solid, you will need to standardize your NaOH solution against a precisely weighed amount of standard acid. The acid used is the weak monoprotic acid, potassium hydrogen. The reaction of KHP with sodium hydroxide is shown below.



KHP Neutralization

PRELAB EXERCISE

Your unknown for this experiment will be a mixture of KHP and an inert material whose composition might be anywhere from 10% to 90% KHP by mass. In order to minimize the contribution of random errors in your analysis, it is important to use a sample size that will require 35-40 mL of titrant for each titration. As in any **real** sample, you do not know the composition, so you must make some assumptions in order to begin the analysis.

If you assume that your unknown is 50% KHP by mass, what mass of unknown should you weigh out in order to require 35 mL of 0.100 M NaOH solution to reach the equivalence point? If the titration of this sample requires only 20 mL of titrant, what must you do to the mass of subsequent samples in order to minimize random errors?

REAGENTS AND APPARATUS

Potassium Hydrogen Phthalate (high purity)
Phenolphthalein indicator solution
50% NaOH solution
50.00 mL buret
Erlenmeyer flasks

PROCEDURE

*** *One week in advance**** You should dry the standard KHP and solid unknown one week in advance. It may also be possible to boil the water in advance.

PART A -- PREPARATION OF 0.1 N SODIUM HYDROXIDE

1. Boil about 1.5 liter of distilled water for 5 minutes to remove carbon dioxide. This will allow you to make enough NaOH solution to do several experiments. Allow the water to cool covered with a watch glass; transfer while warm (40°C) to your large plastic bottle.
2. Using a transfer pipet and a rubber bulb, transfer to this bottle the volume required of a clear saturated, 50%(w/w), solution of NaOH to make the desired concentration of ≈ 0.1 M. The density of the 50%(w/w) NaOH is ≈ 1.50 g/mL. Mix thoroughly and keep covered (*How can atmospheric carbon dioxide lower the concentration of OH?*). Keep this solution even after completion of this experiment, as it will be required for other experiments.

PART B -- STANDARDIZATION OF NaOH SOLUTION

1. Dry 4 to 5 g of primary-standard KHP in a weighing bottle at 110°C for 2 hours. At the same time dry your unknown sample in a separate weighing bottle. Allow to cool 30 minutes in a desiccator before weighing (*why should the desiccator lid be left slightly "cracked" during the cool-down period?*) Save any unused KHP for use later in the semester.
2. On the analytical balance, accurately weigh four samples of the pure, dry KHP standard. Each sample should weigh between 0.6 g - 0.7 g.
3. Dissolve each KHP sample in approximately 100 mL of distilled water in a 250 mL or 500 mL Erlenmeyer flask (*why is the exact volume of water unimportant?*)
4. Add a few drops of phenolphthalein solution.
5. Titrate with the NaOH solution that you are standardizing. The endpoint is faint pink. Strive for consistency in the color and intensity of the endpoint.
6. Calculate the formality of the NaOH for each titration and find the average formality.

PART C -- DETERMINATION OF % KHP IN UNKNOWN

1. Accurately weigh one sample of unknown using the mass calculated in the prelab exercise and dissolve the sample in 100 mL of distilled water in a 250 mL Erlenmeyer flask.
2. Add a few drops of phenolphthalein solution.
3. Titrate with the standardized NaOH to a faint pink endpoint. *NOTE: This titration can be done fairly quickly since it is being used only as a rough estimate of the sample composition.*
4. Based on the results of this titration, determine the optimal sample size to minimize random errors. Accurately weigh four samples of this “ideal” mass of unknown and dissolve each sample in 100 mL of distilled water in a separate 250 mL Erlenmeyer flask.
5. Add a few drops of phenolphthalein and titrate each sample with standardized NaOH to a faint pink endpoint.
6. Calculate the percentage of KHP in the unknown mixture, and its 95% confidence interval.

INFORMATION, HELPS AND HINTS FOR KHP DETERMINATION

Preparation:

- Prepare approximately 1.5 L of 0.100 M NaOH solution. You may find it necessary to use in a future experiment or may also find the time to repeat this experiment in the future. You should be able to predict the amount of 50% NaOH you require for this with knowledge of the fact that we will use a 50% (w/w) solution of NaOH with an approximate density of 1.50 g/mL.
- Both your primary standard KHP and your unknown samples must be dried for at least 2 hours at 110°C. Avoid temperatures much higher than this because the acid may decompose. Once the samples have been dried, store them in your desiccator.
- A point of note, standardization of NaOH and its subsequent use as a titrant are best done within one week of each other. NaOH concentration will change over a period of time, especially if the bottle in which it is contained has been opened and closed many times. One approach that may help is to squeeze excess air out of the bottle before capping it.

Technique:

- Familiarize yourself with correct buret usage. See your text. Remember to estimate volumes to ± 0.01 mL.
- Avoid the bubble that sometimes forms at the tip of the buret. It should be removed before titrating.
- Always record an initial and final buret reading during any titration. Points will be deducted for failure to write both measurements in your notebook.

- It is best to prepare 4 samples each for the standardization and unknown determination. You may then use the first sample to "hone-in" on the approximate volumes of NaOH solution necessary to reach the end point of the titrations. The initial "quick and dirty" titration often actually saves you time. Once it is completed, you can then more quickly add titrant into the other three samples until near the end point and then patiently and dropwise "sneak up" on the end point of the titrations. Even if you prefer not to follow this logic, the fourth sample will allow room for errors or give you a little better statistical relevancy.
- One or two drops of phenolphthalein are sufficient for the titration. Addition of too much indicator will necessitate an **INDICATOR BLANK** (you are strongly advised to look this term up). Also, be certain to learn to be consistent with the addition of amounts of indicator. It will play an important role in later experiments.
- Before progressing to the unknown determination, it is urged that you review your calculated standardization results. If the data are not adequate (i.e. the relative standard deviation of the three triplicate samples used in your standardization should be about 0.25% or less to be considered reasonable), you should repeat these titrations for improved results. Calculations of your results immediately in lab often can save large amounts of setup time because they often allow you to catch problems immediately.

Calculations:

- You may wish to note and correct for the purity of the primary standard KHP (potassium hydrogen phthalate) in your standardization calculations.
- Know how to perform the calculations for standardization and the unknown determination. You will be held responsible for this. **MEMORIZATION OF THE FORMULA IS NOT AN ACCEPTABLE ALTERNATIVE. YOU SHOULD BE ABLE TO DERIVE THE CALCULATION FROM YOUR KNOWLEDGE OF STOICHIOMETRY AND SOLUTION CHEMISTRY.**
- Use the Q-test to evaluate any outliers in your data. See your textbook for details. Remember that well-kept experimental observations can also serve as a subjective means of data evaluation (be sure to document your observations).
- Don't forget the error discussion for the experiment. You will need to evaluate (calculate) the predicted uncertainty for the experiment and to compare it to the standard deviation that you obtain in order to include with this discussion. Not only should you point out possible errors made, but you should also identify the effect of these errors upon your final result.