# Chemistry 212

### Experiment 3

## ANALYSIS OF A SOLID MIXTURE

### LEARNING OBJECTIVES

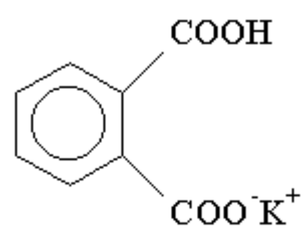
The objectives of this experiment are to

- learn to analyze a solid unknown with volumetric techniques.
- use stoichiometry to determine the percentage of KHP in a solid mixture of KHP and a soluble salt.

### BACKGROUND

In this experiment you will use an acid-base titration to determine the composition of a solid mixture, containing **potassium hydrogen phthalate** (KHP) and an inert, soluble salt (such as NaCl). The analysis will be carried out by weighing a portion of the unknown solid, dissolving it in water, and titrating the resulting solution with a standardized solution of sodium hydroxide.

KHP is an organic weak acid with the structure




which corresponds to formula $KHC_8H_4O_4$. One of the hydrogen atoms in KHP is acidic and thus will react with a base such as $OH^-$. When KHP is dissolved in water, it dissociates to produce potassium ions ($K^+$) and hydrogen phthalate ions ($HP^-$). When $OH^-$ is added to the solution, it extracts the acidic hydrogen atom from the $HP^-$ ion:

$$HP^- + OH^- \rightarrow P^{2-} + H_2O$$

In this experiment, you will titrate the acid solution to the phenolphthalein endpoint. Phenolphthalein is used to help determine the equivalence point of the titration.

The pH of the solution is a measure of the amount of hydrogen ion in the solution:

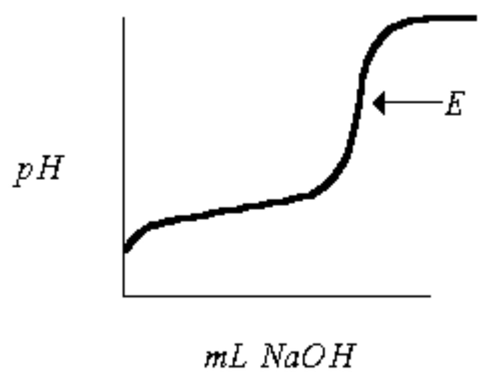
$$pH = -\log [H^+]$$

Hydrogen phthalate is a weak acid:

$$HP^- \Leftrightarrow H^+ + P^{2-}$$

For pure KHP in water, the hydrogen ion concentration will be about $1.0 \times 10^{-4}$ *M* which corresponds to a pH of about 4.0. The value actually observed depends on the concentration of $HP^-$ in solution.

As base is added to a solution of KHP the $HP^-$ will be consumed and the pH of the solution will gradually increase. At first, when there is plenty of $HP^-$ available, the reaction of $H^+$ with base will be offset by more dissociation of $HP^-$, so there will be little change in the pH. This is called the buffering capacity of $HP^-$. But when the $HP^-$ is nearly gone, the change in pH with the addition of $OH^-$ becomes greater. At the point where the $HP^-$ is completely consumed, the change in the pH will be the greatest. Consider the following graph of pH vs. mL of base added, (called a titration curve):




At point E on the curve, the slope of the curve is greatest, since the pH is changing most rapidly in this area of the titration. This is the equivalence point. Location of the equivalence point enables us to determine how many milliliters of base it took to exactly react with the $HP^-$ in solution. Because we also know the concentration of $OH^-$ in the base solution, we can calculate the number of moles and hence grams, of KHP in the original solid sample. The percent KHP (by mass) can then be calculated.

## SAFETY PRECAUTIONS

Safety goggles must be worn in the lab at all times. Any skin contacted with chemicals should be washed immediately.

## EXPERIMENTAL PROCEDURE

Using a standardized NaOH solution (approx. 0.1 M), you will titrate the unknown mixture and determine the percentage of KHP in that mixture.

### Titration of an Unknown Solid Mixture

1. Set up titration apparatus as demonstrated by your instructor.

2. Divide the unknown mixture into four samples of approximately the same size. Dissolve sample #1 (Make sure to record the exact mass from the analytical balance.) of the unknown solid mixture in about 70 mL of deionized water (Use 200 mL beakers). Add several drops of phenolphthalein indicator to the mixture.

3. Titrate the first unknown solution with the standardized NaOH until a slight pinkish color is observed (This is the endpoint of the titration). Make sure to record the beginning and final volume readings from the buret.

4. Repeat with remaining samples.

Scoring will be based on accuracy and precision of your results.

# Chemistry 212

### Experiment 3 – Pre Laboratory Assignment

## ANALYSIS OF A SOLID MIXTURE

1. What is the molar mass of potassium hydrogen phthalate (KHP) See formula above?

2. Calculate the concentration (Molarity) of a solution that contains 2.0 g of KHP dissolved in 100 mL of water.

3. Stoichiometry of Acid-Base reactions.
    a. Define equivalence point.

    b. Define endpoint.

# Chemistry 212

## Experiment 3

# ANALYSIS OF A SOLID MIXTURE

Name(s)____________________________________________

Unknown #__________

| **% KHP in Unknown** | |
|---|---|
| Trial 1 | |
| Trial 2 | |
| Trial 3 | |
| Trial 4 | |
| **Average** | |
| **Std. Dev.** | |

1. Show calculation for Trial 1. (Attach all other notes and calculations.)

2. Discuss the precision of your experiments.

3. Do you feel confident on the accuracy of your results? Explain.