

Name\_\_\_\_\_

Section\_\_\_\_\_

Partner(s)\_\_\_\_\_

Date\_\_\_\_\_

## CHARACTERIZATION OF A MONOPROTIC WEAK ACID BY POTENTIOMETRIC TITRATION

### PRE-LAB QUERIES

1. Complete the neutralization reactions given below:



2. Circle the monoprotic acids in the reactions above.
3. Explain the difference in behavior in aqueous solutions of hydrochloric acid and acetic acid.

### OBJECT

In this activity you will perform an acid-base titration and follow the neutralization reaction by making an electrochemical measurement using a pH meter. From the graphical analysis of the data collected you will determine some characteristics of a weak acid, such as its dissociation constant and molar mass, and how to choose an indicator for visual detection of the end point.

### INTRODUCTION

An electrochemical measurement of the amount of hydrogen ion ( $\text{H}^+$ ) in solution will be accomplished using a combination glass pH electrode and a pH meter or potentiometer. The combination glass pH electrode serves as both the indicator and reference electrodes in one unit. The indicator electrode is the fragile glass membrane (50  $\mu\text{m}$  thick) at the end, which responds to changes in  $\text{H}^+$  concentration by a change in the potential across the membrane. The glass membrane is referenced against a silver-silver chloride electrode of constant potential, connected through a small porous plug on the side of the electrode. To prevent any electrochemical reaction during the measurement (thus a change in concentration of species due to measurement) a potentiometer is used in place of a direct reading voltmeter. The potentiometer measures the cell voltage without drawing current.

In very simple terms, the cell potential (E) is related to the pH of the solution by the following equation:

$$E = E' - (2.303RT/F)\text{pH}$$

where R is the gas constant, T is absolute temperature, F is the Faraday constant, and E' depends on the membrane and internal filling solution. E' is normally constant but will change over long periods of time. The combination glass electrode must be calibrated against a solution of known pH, usually a buffer. The pH meter is set to room temperature to eliminate this as a variable. As a result, the measured E can be read directly in pH units. We will use a computer with a pH probe to follow the titration of a weak acid. This will allow us to measure how the  $[\text{H}^+]$  changes as the neutralization reaction proceeds. From a plot of pH vs. volume of titrant (NaOH) called a titration curve, we can determine the equivalence point or point of complete neutralization. To accurately locate the equivalence point, we will determine the first derivative of the titration curve. The titration curve will provide data to allow us to determine the dissociation constant,  $K_a$ , of the acid and its molar mass.

### PROCEDURE

1. Obtain an unknown solid monoprotic acid sample and record the unknown number. Mass a sample of the solid acid in the range of 0.20 - 0.25 gram to the nearest tenth of a milligram. Place the sample into a 250-mL beaker, add 50 mL of distilled water and stir with a stirring rod. Since most of the solid acid unknowns do not readily dissolve, warm the beaker on a hot plate and, only if needed, add 5 mL increments of ethanol until the sample is completely dissolved.
2. Clean a buret and fill with standard NaOH solution. Record the concentration of the NaOH.
3. On the computer, start the LoggerPro program called **titr.103** (found in the CHM 103 folder). Follow the instruction for a two point calibration using two buffers with set pH values. **Never touch or wipe the glass membrane!!!** Be sure the electrode remains in some solution at all times unless you are transferring or cleaning it.
4. Rinse the electrode with distilled water and place into the dissolved solid acid solution. Record the initial pH of the solution.
5. Place the buret over the beaker to allow smooth delivery of the NaOH. Use a stirring rod to mix the reaction.
6. Start adding titrant in 2-3 mL increments until the pH reaches 4.5, then start adding in smaller increments as the pH changes. Near the equivalence point you will need to decrease the increment to 0.1 mL. Remember you do not want to blow by the equivalence point or you will have poor results! Data should be collected until the curve seems to have leveled off (very little rise with each increment added).
7. Using the program, display the first and second derivatives. These can be used to

accurately determine the equivalence point and, subsequently, the  $K_a$ . Generate graphs which have the original titration curve and each derivative displayed together and print them. Print a complete set of data.

8. Rinse the electrode with distilled water and store as you found it upon completion

### DATA/RESULTS

Unknown solid acid number \_\_\_\_\_

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

Mass of solid acid \_\_\_\_\_

Table of titration data --- attach

Generate and attach the following graphs along with any calculations:

- pH vs. volume of titrant (titration curve.)
- $\Delta\text{pH}/\Delta\text{volume}$  vs. average volume (first derivative of the titration curve)
- $\Delta(\Delta\text{pH}/\Delta\text{volume})/\Delta\text{volume}$  vs average volume (second derivative of the titration curve)

On the titration curve, the equivalence point is where the slope of the curve changes from increasing values to decreasing values. This is called an inflection point by mathematicians. On some titration curves the equivalence point can be difficult to locate due to the gentle change in slope. The first derivative allows easy location of an inflection point (sign of slope changes from positive to negative), since the first derivative will be at a maximum for the equivalence point on a titration curve.

Determine the volume of the equivalence point from the first derivative plot. Locate this volume on the titration curve and label the equivalence point. Record the equivalence point volume and pH in the table that follows.

The second derivative reflects how much change there is in the  $\Delta\text{pH}$  as a function of change in the  $\Delta\text{volume}$ . The second derivative will cross the x-axis ( $y = 0$ ) at equivalence point. Locate this volume on the titration curve and label the equivalence point. Record the equivalence point volume and corresponding pH in the table that follows.

Source	$V_{\text{eq.pt.}}$	$\text{pH}_{\text{eq.pt.}}$
Titration Curve		
First Derivative		
Second Derivative		

The dissociation constant,  $K_a$ , can be obtained from the titration curve. Since the  $\text{p}K_a$  is the pH where the (acid) = (salt) which occurs at  $V_{\text{eq.pt.}}/2$ . Locate it on the titration curve. Calculate the  $K_a$ .

Calculate the molar mass of the weak acid using the equivalence point volume and the sample mass.

For an indicator to work, its color change range ( $\text{p}K_{\text{ind}} \pm 1$ ) must bracket the equivalence point pH. Find two indicators that would correctly work for your solid acid unknown. List the reference source used. Put all information in the table below.

Indicator	Acid color	Base color	pH range
Citation(s)			

## CONCLUSIONS

Summarize what you have determined about the solid acid. Discuss possible sources of error in your process.

## POST-LAB QUESTIONS

1. Compare the shape of a strong acid-strong base titration curve such as HCl with NaOH to the shape of your weak acid-NaOH curve. What general differences in shape occur and why?
2. List three indicators that would work for a strong acid-strong base titration. Cite your information source.
3. A diprotic acid is titrated with NaOH and the first equivalence point volume occurs at 14.32 mL. At what volume will the second equivalence point occur? Why?

