

## Determining the Identity of an Unknown Weak Acid

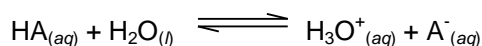
### Purpose

The purpose of this experiment is to observe and measure a weak acid neutralization and determine the identity of an unknown acid by titration.

### Introduction

The purpose of this exercise is to identify an unknown weak acid by titration with a standard sodium hydroxide solution. The pH of the titration solution will be monitored using a pH meter; the obtained titration plot will then be used to determine the equivalent molecular weight and dissociation constant ( $K_a$ ) of the unknown. These values will be used to determine the identity of the weak acid.

When a weak acid (HA) is dissolved in water, only some of the molecules will dissociate to yield  $H_3O^+$  and  $A^-$  ions. At this point, a dynamic equilibrium is established:



Under these equilibrium conditions, the total concentration of each species remains constant, even though the species in solution are constantly dissociating and recombining. The degree of dissociation of the weak acid is used to characterize the acid, and is calculated according to the equation:

$$K_a = \frac{[H_3O^+][A^-]}{[HA]}$$

In the expression,  $K_a$  is the acid dissociation constant. Strong acids typically dissociate completely, and therefore would have a  $K_a$  value of greater than 1. Weak acids have  $K_a$  values much smaller than 1 (typically less than  $10^{-4}$ ). For example, the  $K_a$  of acetic acid (vinegar) is  $1.75 \times 10^{-5}$ , while the  $K_a$  of bicarbonate (baking soda) is  $4.7 \times 10^{-11}$ . For convenience, scientists often use the  $pK_a$  of weak acids, as it allows them to work with whole numbers ( $pK_a = -\log K_a$ ). The  $pK_a$  values of acetic acid and bicarbonate are 4.75 and 10.32, respectively.

When a strong base is added to a solution of a weak acid, the hydroxide ion reacts with some of the  $H_3O^+$  present, therefore disturbing the equilibrium. More of the acid will dissociate, until a new equilibrium is established. When the number of moles of base added equals the number of moles of weak acid present, a sharp change is observed in pH, which can be detected using either a visual indicator or pH meter. This point is the equivalence point, and any additional base added simply increases the pH. This information can be used to determine the quantity (in moles) of acid that is present.

The volume and concentration of the added base can be used to determine the number of moles of acid present (by assuming a 1:1 molar ratio of acid:base). By measuring the pH of solution after each addition of base, a titration curve can be constructed. The titration curve allows for the determination of the  $K_a$  value of the acid. According to the equation above,  $K_a$  will be equal to  $[H_3O^+]$  when  $[A^-] = [HA]$ , and the  $pK_a$  will equal the pH at this point. This condition is satisfied halfway to the equivalence point of the titration.

In this lab, the identity of an unknown weak acid will be determined by constructing a titration curve, estimating the value of  $pK_a$ , and determining the molar mass of the acid based on the quantity of standard solution required.

Adapted from R. C. Kerber et. al [http://www.sinc.sunysb.edu/Class/orgolab/che199\\_susb014.PDF](http://www.sinc.sunysb.edu/Class/orgolab/che199_susb014.PDF); W.F. Kinard et.al <http://www.cofc.edu/~kinard/221LCHEM/2002CHEM221LabSchedule.htm>; and D.C. Harris Quantitative Chemical Analysis, 5<sup>th</sup> ed Freeman Press, 1998.

## Determining the Identity of an Unknown Weak Acid

### Procedure

Remember to record all measurements and unknown numbers on your data tables. Record buret volumes to the nearest 0.01 mL.

1. Dissolve between 1.3 and 1.4 grams of the unknown acid sample in a 100-mL volumetric flask, using 0.10 M KCl solution. If it does not dissolve easily, you may *gently* heat the mixture. Allow this solution to cool to room temperature before titrating.
2. Assemble a titration setup using a buret, ring stand and buret clamp; obtain a stir plate and place it under the buret. Rinse the buret with a few 3 mL portions of base before filling with NaOH solution.
3. Set up and calibrate the pH meter. Remember to store the electrode in the provided buffer solution when not in use.
4. Use a volumetric pipette to transfer 25 mL of the acid solution to a clean 100 mL beaker and place this beaker on the stir plate. Add a stir bar and place the pH electrode in the acid solution, making sure the tip is completely immersed. Make sure that the stir bar does not hit the electrode. Record the initial pH and initial buret reading.
5. With stirring, add 2-mL portions of base to the acid solution, recording the cumulative (total) volume of base added and the pH after each addition.
6. When the pH begins to significantly change (by ~ 0.2 units), decrease the volume of base added to 0.1 – 0.2 mL portions, as the titration proceeds through the equivalence point. Using smaller increments of base will improve the equivalence point estimation.
7. Once through the equivalence point, the volumes of base added can be increased to 2-mL portions. Continue the titration at least 10 mL past the equivalence point.
8. Repeat steps 4-7 for trial 2.

### Clean-Up

Wash all glassware with soap then rinse 3 times with tap water, and once with deionized water.

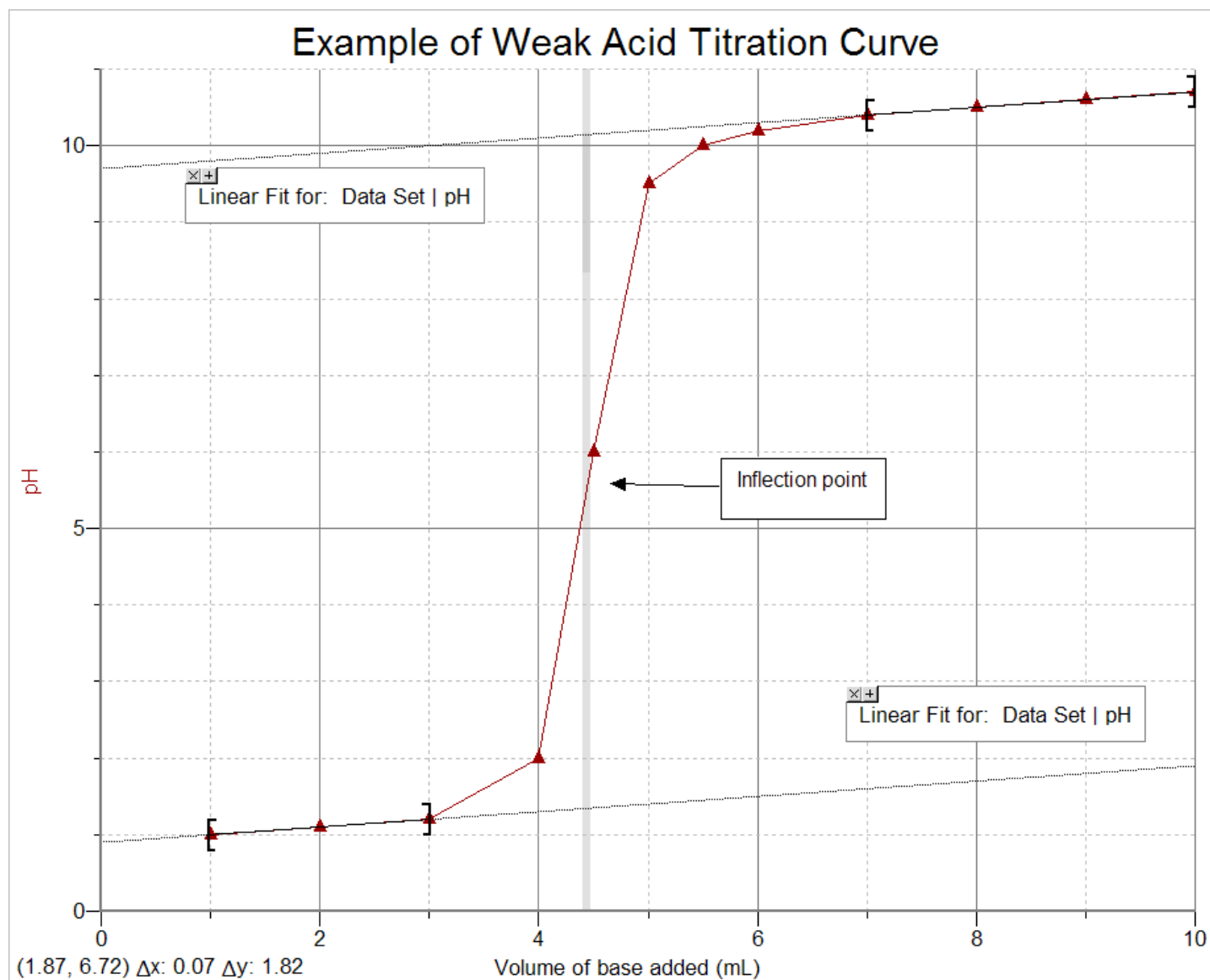
### Data Analysis and Calculations

Use the data collected to construct titration curves of pH (y-axis) versus volume of base added (x-axis) for each trial. Although using graph paper and plotting the points will certainly work, your instructor may wish for you to use a spreadsheet program such as *Microsoft Excel* or a graphing program such as *Graphical Analysis*. Remember to label axes and give each graph a unique title.

The equivalence point of the titration is defined as the point of inflection (where the slope changes) on the titration curve. The  $pK_a$  of the acid can be estimated by reading the pH value at  $\frac{1}{2}$  equivalence volume ( $V_e$ ) on the titration curve.

To determine the inflection point look at your graph of pH versus volume added. You should be able to see a series of points in the beginning of the titration that would make more or less a straight but not flat line and a series of points at the end of the titration that would also make more or less a straight but not flat line. Use a ruler to make lines connecting each set of points. Extend the lines past the points themselves. The lines should be almost parallel to each other. Take a ruler and measure the distance between these lines. Then (keeping the ruler perpendicular to both lines as best you can) mark off on the steeply rising portion of the graph the midpoint using half of the distance between the lines. Your instructor will demonstrate this method in class. See the following diagram.

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Calculate the molar mass of the unknown acid using the mass of acid titrated and moles of NaOH added.

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### Weak Acid Unknown Possibilities

Use the following table to choose the identity of your unknown acid based on the calculated molecular weight and dissociation constant values, pKa values taken

Compound	Formula	Molar Mass	pKa
Acetic acid	HC <sub>2</sub> H <sub>3</sub> O <sub>2</sub>	60.0	4.756
Propanoic acid	HC <sub>3</sub> H <sub>5</sub> O <sub>2</sub>	74.1	4.874
Crotonic acid	HC <sub>4</sub> H <sub>5</sub> O <sub>2</sub>	86.1	4.676
d/l-lactic acid	HC <sub>3</sub> H <sub>5</sub> O <sub>3</sub>	90.1	3.858
Chloroacetic acid	HC <sub>2</sub> ClH <sub>2</sub> O <sub>2</sub>	94.5	2.867
Potassium hydrogen oxalate	KHC <sub>2</sub> O <sub>4</sub>	128.5	4.272
Potassium bisulfate	KHSO <sub>4</sub>	136.2	1.99
d/l-mandelic acid	HC <sub>8</sub> H <sub>7</sub> O <sub>3</sub>	152.1	3.411
Sulfanilic acid	HC <sub>6</sub> H <sub>6</sub> NO <sub>3</sub> S	173.2	3.738
Potassium hydrogen tartrate	KHC <sub>4</sub> H <sub>4</sub> O <sub>6</sub>	188.1	4.366
Potassium hydrogen phthalate	KHC <sub>8</sub> O <sub>8</sub>	204.2	5.408

from *Lange's Handbook of Chemistry*, 13<sup>th</sup> edition









## Determining the Identity of an Unknown Weak Acid Data Sheets

Name: \_\_\_\_\_

Lab Partner: \_\_\_\_\_

**Unknown #:** \_\_\_\_\_

Mass of acid used (g)	
Total volume of prepared acid sample (mL)	
Concentration of prepared acid sample (g/mL)	
Concentration of base	

	Trial 1	Trial 2
Volume of acid titrated (mL)		
Mass of acid titrated (g)		
Equivalence Volume ( $V_e$ ) (inflection point from curve)		
Moles of base required for equivalence		
Molar mass of unknown acid (assume 1:1 stoichiometry)		
$pK_a$ of unknown acid (pH at $\frac{1}{2} V_e$ – from curve)		

**Average Molar Mass:** \_\_\_\_\_**Average  $pK_a$ :** \_\_\_\_\_**Identity of Unknown Acid (use table):** \_\_\_\_\_

Note: If the experimental molar mass and  $pK_a$  values do not both agree with the table on page 3, generally a molar mass comparison will give a more reliable identification of the weak acid.

**Calculations:** Show all work on separate paper as necessary.



## Determining the Identity of an Unknown Weak Acid Pre-Lab Questions

Name: \_\_\_\_\_

1. In your own words, define equivalence point:
2. How do a weak acid and strong acid differ? Give examples of strong and weak acids.
3. What ionic species are produced when a weak acid reacts with water?
4. Write the complete and net-ionic equation for the neutralization of acetic acid by sodium hydroxide.
5. The value of  $K_a$  in water at 25 °C for propionic acid ( $C_2H_5CO_2H$ ) is  $3.4 \times 10^{-5}$  M. What is the pH of a 0.020 M aqueous solution of propionic acid?