Characterization of Weak Acids

INSTRUCTOR RESOURCES

The CCLI Initiative

Learning Objectives

The objectives of the experiment are to . . .

- understand the titration curve for a weak acid.
- calculate the molar mass of the weak acid.
- calculate the dissociation constant for the weak acid.
- use the molar mass and values to identify the acid.

Procedure Overview

- after calibration of drop size and the pH electrode, a solution of NaOH is standardized with KHP.
- an unknown weak acid is titrated with the standardized base. The molar mass and dissociation constant for the acid are calculated.
- the identity of the unknown acid is determined from the molar mass and Ka values.

Name _____ Section ____ Date _____

CHARACTERIZATION OF A WEAK ACID

Report Sheet

Calibration of drop size

Give the equation for conversion from drops NaOH to ml NaOH:

Standardization of NaOH

	Trial 1	Trial 2	
data file name			
mass KHP	g	g	
molarity NaOH	M	M	
	Average molarity	M	

Titration of unknown acid

Unknown #				
	Trial 1	Trial 1	Trial 1	Trial 1
data file name				
mass of acid	g	g	g	g
volume H2O	ml	ml	ml	ml
molar mass of acid				
		Aver	age molar mass	
Kal values				
Ka2 values				
	Average	Ka1	Ka2	
Identity of unknown acid				

Name _____ Section ____ Date _____

CHARACTERIZATION OF A WEAK ACID

Report Sheet (page 2)

Calculations

Standardization of NaOH

1. Determine the moles of KHP.

2. Calculate the molarity of NaOH for each trial.

3. Calculate the average molarity of NaOH.

Titration of unknown acid

- 1. Calculate the molar mass for the acid.
- 2. Determine the dissociation constant(s) for the acid at a minimum of *three* points along the curve.

Name	Section	Date

Questions/Problems

1. A 33.50 ml volume of 0.1050 *M NaOH* was required to just neutralize a 0.2500 g sample of a monoprotic organic acid dissolved in 50.00 ml of water. What is the molar mass of the acid? If the dissociation constant of the acid were 3.0 x 10-6, what would be the pH

- 2. Indicate how each of the following would affect the values of and molar mass for a weak acid calculated in this experiment. (In other words, would the Ka and molar mass obtained be higher, lower, or unchanged from what it should be?)
 - a. the molarity of the NaOH is actually greater than believed.

- b. the pH meter reads 0.60 high all the time.
- c. the sample of weak acid was actually impure, containing about 5% of an inert material.

Name	Section	Date

Questions/Problems (page 2)

- 3. A 0.0050 mole sample of a weak acid with a dissociation constant of $1.0 \ge 10-7$ was titrated with 0.200 *M* NaOH, after being dissolved in 50.0 ml of water. Calculate the pH of the solution.
 - a. before any NaOH was added.

b. after 10.0 ml of NaOH was added.

c. after 24.9 ml ofNaOH was added.

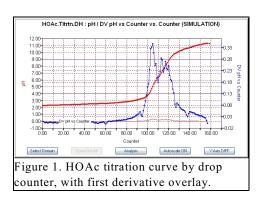
d. after 25.1 ml ofNaOH was added

Tips and Traps

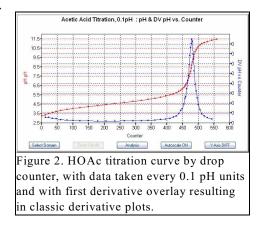
- 1. Students must use boiled, deionized water for their solutions.
- 2. It is helpful to show students the proper titration set-up. The tip of the buret should be level with the top of the drop counter and centered on the cross lines on the case for optimum results.
- 3. Students should take time to align the drop counter properly. Most problems in the titrations result from a poorly aligned counter. No program is necessary for alignment. All students need to do is start the buret dripping and watch the counter light on the interface. If it blinks at each drop, alignment is correct.
- 4. The KHP should be dried at 110°C for two hours.
- 5. Unknown acids should NOT be dried. Some of them will decompose.
- 6. It is easiest to take all data for all trials at one sitting instead of making one solution at a time.

Using the *MicroLAB* tools

Instructions on the use of the *MicroLAB* tools such as probe calibration, use of the drop counter, obtaining derivatives and interpolating between titration points are given in **Useful Titration Operations** within the **Useful Tools folder**. Their first derivative graphs may look similar to the



following graph. There is a lot of noise around the equivalence point which results from а s m a l l irregularities in the pH caused by mixing of the solution and the lag time in the probe to measure pH. When the data is read



every 0.1 pH units, it produces a classic first and second

derivative as seen in Figure 2. That peak point is closest to the equivalence point. The second derivative at that point, going from positive to negative, will then give the exact equivalence point. This then gives the volume of titrant required to reach the equivalence point, from which can be calculated the number of moles of the analyte.

Tips and Traps

Titration Program: The following program will result in the type of titration curve and derivative as seen in the figure.

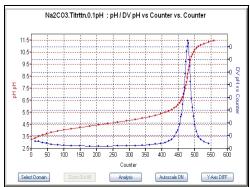
Acid/Base Titration, data taken every 0.1 pH units. Use for any titration of an acid with a base, or a base with an acid, recording the data each time the pH has changed by 0.1 pH units. Use of this program results in very smooth titration curves and generally very well shaped derivatives. Experiment name: . *pH.temp.vs.drop.titr.0.1pH.exp*.

Sensors: **drop counter**: X axis, Col. A, DD on top, units = drops; **pH**: Y1 axis, Col B, DD in middle, units = pH; **Temp**: Y2 axis, Col C, DD on bottom, units °C. (Use of temperature at instructors discretion.)

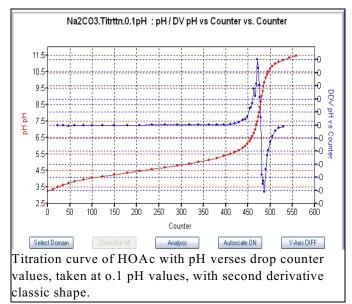
Special Program:

Read Sensors Repeat when counter change (Sets to read only when a drop has passed through the counter.) If Delta pH > +/- 0.100 (Sets to read only when pH has changed by 0.1 pH units.) Read Sensors (Reads all variables selected in Data Sensors/Variables and stores in a data grid.) Else End If Until Stop Button is pressed

Comment: Calibrate the drop counter using one of the *dropcal.exp* before and after the series of titrations . If temperature is measured with a Temp(IC) probe, it must be wrapped in Saran Wrap to prevent grounding the pH probe. This program can be saved as a **Template**. See **Measurement Manual**



Titration curve of HOAc with pH versus drop counter values. In this titration the pH was taken only after the pH had changed by 0.1 pH units. Note that the curve is much smoother, and the first derivative is classic in shape.



for details.

Suggested Answers to Questions/Problems

1. A 33.50 ml volume of 0.1 050 M NaOH was required to just neutralize a 0.2500 g sample of a monoprotic organic acid dissolved in 50.00 ml of water. What is the molar mass of the acid? If the dissociation constant of the acid were 3.0 x 10-6, what would be the pH at the equivalence point?

Molar Mass = 71.07 g *pH*=3.45

- 2. Indicate how each of the following would affect the values of and molar mass *for* a weak acid calculated in this experiment. (In other words, would the Ka and molar mass obtained be higher, lower, or unchanged *from* what it should be?)
 - a. the molarity of the NaOH is actually greater than believed.

Ka: lower MM: higher

b. the pH meter reads 0.60 high all the time.

Ka: higher MM: unchanged

c. the sample of weak acid was actually impure, containing about 5% of an inert material.

Ka: lower MM: higher

- 3. A 0.0050 mole sample of a weak acid with a dissociation constant of 1.0 x 10-7 was titrated with 0.200 M NaOH, after being dissolved in 50.0 ml of water. Calculate the pH of the solution.
 - a. Before any NaOH was added. pH = 4.00
 - b. After 10.00 ml of NaOH was added pH = 6.82
 - c. After 24.9 ml of NaOH was added pH = 9.40
 - d. After 25.1 ml of NaOH was added *pH= 10.42*

Sample Data

Calibration of drop size Students should provide the equation for conversion from drops NaOH to ml NaOH:

Standardization of NaOH

mass KHP	Trial 1 0.5910 g		Trial 2 0.6020 g	
data file name molarity NaOH	0.09516 M Average molarity		0.09482 M 0.049499 M	
Titration of unknown acid				
Unknown #				
	Trial 1	Trial 1	Trial 1	Trial 1
mass of acid	0.3937 g	0.3152 g	0.3843 g	0.3521 g
volume H2O	ml	ml	ml	ml
data file name				
molar mass of acid	150.9	152.1	152.7	151.9
	Avera	ge molar mass i	151.9 (Actua	al 152.15)
Kal values	3.9 x 10 ⁻⁴	3.5 x 10 ⁻⁴	3.5 x 10 ⁻⁴	
Ka2 values				
	Average	Ka13.6 x 10 ⁻⁴	Ka2	(actual 1.4 x 10 ⁻⁴)
Identity of unknown acid	Mandelic acid			

Calculations

Calculations

Standardization of NaOH

1. Determine the moles of KHP.

$\frac{0.5910 \text{ g KHP}}{204.3 \text{ g/mol}} = 2.893 \text{ xl0}^{-3} \text{ mol KHP}$

2. Calculate the molarity of NaOH for each trial.

$$\frac{2.893 \text{ X } 10^{-3} \text{ mol OH}^{-}}{30.41 \text{ x } 10^{-2} \text{ L}} = 0.09516 M$$

3. Calculate the average molarity of NaOH.

$$\frac{0.09516 M + 0.09491 M + 0.09474 M}{3} = 0.9499 M$$

Titration of unknown acid

1. Calculate the molar mass for the acid.

475 x 0.05784 ml x 0.09499 mmol = 2.6098 mmol drop ml

 $\frac{0.3937 \text{ g}}{2.6098 \text{ x } 10^{-3} \text{ mol}} = 150.9 \text{ g/mol}$

2. Determine the dissociation constant(s) for the acid at a minimum of *three* points along the curve.

Varies with unknown . . .

Laboratory Preparation (per student station)

Equipment

- pH electrode
- ring stand
- *MicroLAB* drop counter
- buret with buret clamp
- Nalgene bottle
- wash bottle (for NaOH)
- 250 ml beakers

Supplies

• paper towels

Chemicals

- KHP
- unknown acids (2.5 2.6 g samples), see attached list
- sodium hydroxide stock solution (6 M)
- buffer solution (pH 7.0)

Safety and Disposal

• no special precautions needed

Unknown Acids (Use 2.5 - 2.6 gram samples)

• adipic acid

tartaric acid

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•	benzoic acid	a-bromoacetic acid
•	o-bromobenzoic acid	a-chloroacetic acid
•	o-chlorobenzoic acid	3-chloropropanoic acid
•	p-chloropropanoic acid	1 1
•	dimethylmalic acid	fumaric acid
•	p-hydroxybenzoic acid	lactic acid
•	mandelic acid	maleic acid
•	malonic acid	
•	o-nitrobenzoic acid	
•	oxalic acid	
•	phenylacetic acid	phthalic acid
•	potassium hydrogen phosphate	potassium hydrogen phthalate
•	salicylic acid	succinic acid
•	sulfanilic acid	

trimethylacetic acid