

## **PRE-LAB FOR EQUILIBRIUM LAB**

### **PART I. TITRATION OF AN AMINO ACID USING A pH METER**

### **PART II. SPECTROPHOTOMETRIC STUDY OF AN INDICATOR**

#### **GOAL/PURPOSE:**

- to monitor pH during an acid/base titration using a pH meter
- to determine the molecular weight of an unknown amino acid
- to use a spreadsheet program (Quattro Pro) to graph the titration curve
- to explore the relationship between pH and acid/base indicator color using a UV/vis spectrophotometer (diode array)

#### **NEEDED PRIOR CONCEPTS:**

- acid/base equilibria
- titration
- acid/base indicators
- pH/pK<sub>a</sub> and relative acid strength
- visible spectroscopy
- using titration to determine equivalent weight, and molecular weight of an unknown acid
- amino acids and functional groups
- Zwitter ions

\*\* the lab could be simplified/made qualitative by eliminating the "unknown" acid aspect of determining the molecular weight of the amino acid. This reduces the need for discussion of pK<sub>a</sub>. Alternatively, this could be done after a preliminary experiment in which a student determines, for example, the K<sub>a</sub> for acetic acid using indicators (methyl red and bromcresol green work fairly well).

#### **LABORATORY SKILLS:**

- analytical balance
- pH meter
- titration/use of a buret
- micropipettors
- UV-Visible diode array spectrophotometer
- use of spreadsheet programs

#### **PRE/POST -LAB ACTIVITIE:**

- use of Spectronic 20 spectrophotometer; comparison with diode array spectrophotometer
- relate amino acids to protein synthesis
- pre-lab: simple titration of acid/base using an indicator
- intro: usefulness in identification of amino acids; relate to problems in metabolism of phenylalanine
- post-lab extension: red wine, rose petals, red cabbage investigation of spectra at different pH's; compare to indicator used in lab: one molecule or more present?

### **SAFETY:**

- stirrer used as stirrer only, not hot plate
- care of pH electrode
- stool for use as needed in reading of buret during titration
- care and handling of cuvettes for spectrophotometer
- micropipettors: take care not to invert; label pipettes for use with specific solutions

### **PROCEDURES:**

#### **Part I**

1. Video: analytical balance
2. Caution not to turn on hot plate
3. Video: Apparatus set up and operation
4. Video: Operation of pH meter; standardization
5. Caution on speed of addition of liquid from buret: dropwise

#### **Part II**

1. Video: micropipettor use
2. Check off solutions as they are added in order not to omit one in error.
3. Video: UV/Vis spectrophotometer
4. Care of cuvettes

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## POST-LAB ACID-BASE EQUILIBRIUM

1. Define equivalence point.
2. Consider the following titration of a weak acid with a strong base, which is what was done in the experiment you just completed. Suppose we titrated 100.0 mL of 0.100 M acetic acid ( $\text{CH}_3\text{COOH}$ ) with 0.100 M NaOH. ANSWER THE FOLLOWING SHOWING YOUR CALCULATIONS:
  - a. What would be the pH of the acetic acid before any base is added? ( $K_a = 1.8 \times 10^{-5}$ )
  - b. What would be the pH after 20.0 mL of NaOH has been added?
  - c. What would be the pH after 40.0 mL of NaOH has been added?
  - d. What would be the pH after 50.0 mL of NaOH has been added?
  - e. What would be the pH after 60.0 mL of NaOH has been added?
  - f. What would be the pH after 99.0 mL of NaOH has been added?
  - g. What would be the pH after 100.0 mL of NaOH has been added?
  - h. What would be the pH after 101.0 mL of NaOH has been added?
  - i. What would be the pH after 110.0 mL of NaOH has been added?
3. Using the values for pH that you calculated in #2, make a plot of pH vs. mL of NaOH added.
4. Looking at your graph, where would you determine the pH of the equivalence point to be?
5. At what pH would the acetic acid have the best buffering capacity?
6. What is the role an indicator plays in titration?
7. What type of chemical are indicators?
8. In a titration, an indicator must be chosen using what criteria?
9. What is the difference between the equivalence point and the end point?

POST-LAB/EQUILIBRIUM/ANSWER KEY

1. The equivalence point is the point in a titration when enough titrant has been added to react completely with the substance being titrated.

2a.

$$K_a = \frac{[H^+][CH_3COO^-]}{[CH_3COOH]} = \frac{(x)(x)}{0.100-x} = \frac{x^2}{0.100} = 1.8 \times 10^{-5}$$

$$x = 1.3 \times 10^{-3} \quad [H^+] = 1.3 \times 10^{-3} \quad pH = -\log(1.3 \times 10^{-3}) = 2.89$$

2b.

Moles NaOH to react:  $20.0 \text{ mL}(0.100 \text{ mol}/1000\text{mL})=2.00 \times 10^{-3} \text{ mol NaOH}$

Moles  $CH_3COOH$  before reaction:  $100.0\text{mL}(0.100 \text{ mol}/1000 \text{ mL})=0.010 \text{ mol } CH_3COOH$

$$K_a = \frac{[H^+][CH_3COO^-]}{[CH_3COOH]} = \frac{(x)(2.00 \times 10^{-3}/0.120 \text{ L})}{0.010 - (2.00 \times 10^{-3}/0.120 \text{ L})} = 1.8 \times 10^{-5}$$

$$x=7.2 \times 10^{-5} \text{ M} \quad pH = -\log(7.2 \times 10^{-5}) = 4.14$$

2c.

$40.0 \text{ mL } (0.100 \text{ mol}/1000\text{mL})=4.00 \times 10^{-3} \text{ mol NaOH}$

$0.010 \text{ mol } CH_3COOH$

$$K_a = \frac{(x)(4.00 \times 10^{-3}/0.140 \text{ L})}{((0.010-4.00 \times 10^{-3})/0.140 \text{ L})} = 1.8 \times 10^{-5}$$

$$x=2.7 \times 10^{-5} \text{ M} \quad pH = -\log(2.7 \times 10^{-5})=4.57$$

2d.

$50.0 \text{ mL}(0.100 \text{ mol}/1000\text{mL})=5.00 \times 10^{-3} \text{ mol NaOH}$   $0.010 \text{ mol } CH_3COOH$

$$K_a = \frac{(x)(5.00 \times 10^{-3}/0.150 \text{ L})}{((0.010-5.00 \times 10^{-3})/0.150 \text{ L})} = 1.8 \times 10^{-5}$$

$$x=1.8 \times 10^{-5} \text{ M} \quad pH=4.74$$

2e.

$60.0 \text{ mL}(0.100 \text{ mol}/1000\text{mL})=6.00 \times 10^{-3} \text{ mol NaOH}$   $0.010 \text{ mol } CH_3COOH$

$$K_a = \frac{(x)(6.00 \times 10^{-3}/0.160 \text{ L})}{((0.010 - 6.00 \times 10^{-3})/0.160 \text{ L})} = 1.8 \times 10^{-5}$$

$$x = 1.2 \times 10^{-5} \text{ M} \quad \text{pH} = 4.92$$

$$2f. \quad K_a = \frac{(x)(9.90 \times 10^{-3}/0.199 \text{ L})}{((0.010 - 9.90 \times 10^{-3})/0.199 \text{ L})} = 1.8 \times 10^{-5}$$

$$x = 1.8 \times 10^{-7} \text{ M} \quad \text{pH} = 6.74$$

2g. *Equivalence point*

$$K_b = K_w/K_a = 1.0 \times 10^{-14}/1.8 \times 10^{-5} = 5.6 \times 10^{-10} = [\text{OH}][\text{CH}_3\text{COOH}]/[\text{CH}_3\text{COO}^-]$$

$$(x^2)/(0.010 \text{ M} - x) = 5.6 \times 10^{-10}$$

$$x = 2.4 \times 10^{-6} \quad \text{pH} = 14.00 - 5.63 = 8.37 \quad \text{pOH} = -\log(2.4 \times 10^{-6}) = 5.63$$

$$2h. \quad 101.0 \text{ mL}(0.100 \text{ mol}/1000 \text{ mL}) = 0.0101 \text{ mol NaOH}$$

*OH from H<sub>2</sub>O will be negligible*

$$0.0101 \text{ mol} - 0.0100 \text{ mol} = 0.0001 \text{ mol OH excess}$$

$$[\text{OH}^-] = 0.0001 \text{ mol}/0.201 \text{ L} = 5.0 \times 10^{-4} \text{ M}$$

$$\text{pOH} = 3.3 \quad \text{pH} = 10.7$$

$$2i. \quad 110.0(0.100 \text{ mol}/1.0 \text{ L}) = 0.011 \text{ mol NaOH}$$

*Again, OH from H<sub>2</sub>O will be negligible*

$$0.0110 - 0.0100 \text{ mol} = 0.001 \text{ mol OH excess}$$

$$[\text{OH}^-] = 0.001 \text{ mol}/0.210 \text{ L} = 4.8 \times 10^{-3}$$

$$\text{pOH} = 2.32 \quad \text{pH} = 11.7$$

3.

$$4. \quad \text{pH} = 8.37$$

$$5. \quad \text{pH} = 4.57$$

6. *It changes color to let you know you are near the equivalence point.*
7. *They are weak acids or bases that change slightly and this alters their optical properties.*
8. *The pH of the equivalence point should fall in the range for the color change of an indicator.*
9. *The endpoint is where the indicator changes color. The equivalence point is where equal amounts of acid and base have been added. (With proper selection of indicators the error between the two will be small.)*

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**TOPS Lab: Equilibrium Part II**

Names \_\_\_\_\_

**Spectrophotometric Study of an indicator**

Follow the directions for each question/section. Answer in the space provided.

1. Find a chart of the Electromagnetic Spectrum in your text. Look at all the labels given for the chart. What quantity (wavelength, energy, frequency, etc.) is increasing from left to right in this figure?

2. Which type of light (electromagnetic radiation) is more damaging to your skin: ultraviolet or microwaves?

Use your answer for Question #1 to explain why this is true.

3. List the colors in the visible spectrum from high energy to low energy.

What types of light can be found in sunlight?

Questions #5-10 refer to the figure on the other side of this sheet.

5. Study the chart on the other side of this sheet. What quantity is increasing from left to right on this chart?

6. What, if anything would you have to do to the chart on the other side of this sheet to make it coincide with, or read the same way as the figure in your textbook?

7. Which type of light is more energetic, red or blue visible light?

8. How is the energy of the light related to the wavelength?

9. Which of these figures, the one given here or the one in your textbook, matches or is similar to the spectrophotometric graph you obtained in the lab? (If both are similar, indicate why you think so.)

Questions 10-20 refer to the graph you printed out in the experiment.

10. Label the curves on the graph using A,B,C designations as well as the actual color and pH of each solution.

Identify the pH range of acid solutions \_\_\_\_\_, basic solutions \_\_\_\_\_, neutral solutions \_\_\_\_\_ .

11. Identify the quantity plotted on the horizontal axis of your graph.

Does it increase or decrease from left to right?

Write the names for the colors of the visible spectrum in the proper region on the horizontal axis of the graph.

12. Explain the difference between the terms "absorbed" and "transmitted". Use a dictionary if necessary.

13. If the absorbance line goes up at a particular color region of the spectrum, what does that mean in terms of how the material will look? (color it will appear to be).

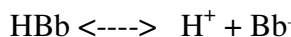
14. If the bromthymol blue was visually blue, what region in the visible portion of the spectrum (color of light) is absorbed by the solution?

15. If the bromthymol blue solution is visually yellow, what portion of the spectrum (color of light) is transmitted by the solution?

16. What color is bromthymol blue in

- (a) acid solution \_\_\_\_\_
- (b) basic solution \_\_\_\_\_
- (c) neutral solution \_\_\_\_\_

17. Acid-base indicators, such as bromthymol blue, can be thought of as weak acids themselves, i.e. as molecules which gain or lose a  $H^+(aq)$  in acid-base reactions. In acid solution there are lots of  $H^+(aq)$  around and the molecule is in the form "HBb" (where Bb represents the rest of the bromthymol blue molecule other than the proton,  $H^+$ , which is gained or lost.) In basic solution, the  $H^+$  is pulled off the molecule, and it is in the form Bb-. The reaction can be represented as:



color: \_\_\_\_\_ colorless \_\_\_\_\_

Identify the color of each of the two forms of bromthymol blue by filling in the blanks above. (The double arrow indicates an "equilibrium" reaction. It can go either way, depending on the conditions.)

18. What is the color of bromthymol blue in neutral solution?

Consult with other lab groups as needed. How do you account for the color? Explain your answer fully.

19. Suppose you made errors in using the pipette and pipette pump (e.g. disregarding the graduation marks and draining the pipette completely), thus adding more than 10 mL indicated on the pipette. How would this affect the color of your solution and the results graphed by the spectrophotometric trace? Explain your answer fully.

20. Write a statement describing the relationship between pH and the concentration of a colored ion in solution.