**EXPERIMENT 7**

Preparation of Buffers

**INTRODUCTION**

A buffer solution contains a weak acid and its conjugate base, and the solution is resistant to pH changes. If a strong base is added to a buffer solution, the weak acid reacts with the added base, allowing the pH of the solution to remain virtually unchanged. If a strong acid is added to a buffer solution, the conjugate base reacts with the added acid, allowing the pH of the solution to remain virtually unchanged.

In the preparation of an effective buffer, there are two important considerations.

1. **CONCENTRATIONS OF THE WEAK ACID AND CONJUGATE BASE.** The concentration of the buffering solutes (the weak acid and its conjugate base) are usually in the range of 0.1 to 0.5 molar, although there are media in which the concentrations are much lower. Very dilute buffer solutions can readily be “overwhelmed” by the addition of strong acids or bases. This is often the effective action that changes the colors of acid-base indicators.

2. **RATIO OF WEAK ACID TO CONJUGATE BASE.** The ratio of the weak acid concentration to the conjugate base concentration is usually between 1:10 and 10:1 for the most effective buffering action. Substituting these ratios into the Henderson-Hasselbach equation:

\[
pH = pK_a + \log \frac{[A^-]}{[HA]}
\]

gives us a pH range over which a buffer solution is most effective, that is from

\[
pH = pK_a + \log(1/10)
\]
\[
= pK_a + \log(0.1)
\]
\[
= pK_a - 1
\]

to

\[
pH = pK_a + \log(10/1)
\]
\[
= pK_a + \log(10)
\]
\[
= pK_a + 1
\]

This means that to prepare an effective buffer of a specific pH, the weak acid used should have a \(pK_a\) value that is within 1 unit of the desired pH.
The Henderson-Hasselbach equation is useful in preparing buffer solutions. In a buffer solution, because the ratio of the moles of conjugate base to the moles of weak acid is equal to the ratio of the concentration of conjugate base to the concentration of weak acid, a substitution that is often helpful is:

$$pH = pK_a + \log \left( \frac{n_{A^-}}{n_{HA}} \right)$$

There are three methods to prepare simple buffer solutions.

1. **Mixing a Weak Acid and Its Conjugate Base.** If one has both a weak acid solution and a solution of its conjugate base, directly mix them together will produce a buffer. To produce a buffer of a specific pH, the Henderson-Hasselbach equation can be used along with the desired pH and the $pK_a$ of the weak acid. The components necessary to produce the buffer can be calculated from the following:

$$pH - pK_a = \log \left( \frac{n_{A^-}}{n_{HA}} \right)$$

2. **Mixing a Weak Acid and a Lesser Amount of a Strong Base.** If one has a weak acid solution, one can add a strong base such as NaOH as the limiting reactant, thereby neutralizing a portion of the weak acid and producing the conjugate base. The components necessary to produce the buffer can be calculated from the following:

$$pH - pK_a = \log \left( \frac{n_{A^-} + n_{OH^-}}{n_{HA} - n_{OH^-}} \right)$$

3. **Mixing a Weak Base and a Lesser Amount of a Strong Acid.** If one has a solution of a weak base, one can add a strong acid such as HCl as the limiting reactant, thereby neutralizing a portion of the weak base and producing the conjugate acid. The components necessary to produce the buffer can be calculated from the following:

$$pH - pK_a = \log \left( \frac{n_{A^-} - n_{H^+}}{n_{HA} + n_{H^+}} \right)$$

In this experiment you will (1) investigate the different methods for preparing buffers; (2) practice choosing suitable buffer solutions for given pH’s, and (3) test buffering capacity and effective buffering range by adding HCl and NaOH to a buffer.

**PROCEDURE**

1. Students will work individually for this experiment. Except for the laboratory handout, remove all books, purses, and such items from the laboratory bench top, and placed them in the storage area by the front door. For laboratory experiments you should be wearing closed-toe shoes. Tie back long hair, and do not wear long, dangling jewelry or clothes with loose and baggy sleeves. Open you lab locker. Put on your safety goggles, your lab coat, and gloves.
2. The following solutions will be available to produce the buffer solutions in this experiment,

<table>
<thead>
<tr>
<th>Chemicals</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1000 M HCl (found on the side counter)</td>
</tr>
<tr>
<td>1.000 M NaOH</td>
</tr>
<tr>
<td>0.40 M NH₄Cl</td>
</tr>
<tr>
<td>0.40 M NaH₂PO₄</td>
</tr>
<tr>
<td>Solid NaC₂H₃O₂·3H₂O</td>
</tr>
<tr>
<td>0.1 M NaH₂PO₄/0.1 M Na₂HPO₄ buffer (prepared by stockroom)</td>
</tr>
</tbody>
</table>

and the following data concerning the available acids will be valuable.

<table>
<thead>
<tr>
<th>Acid</th>
<th>$K_a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>H₃PO₄</td>
<td>7.6 x 10⁻³</td>
</tr>
<tr>
<td>H₂PO₄⁻</td>
<td>6.2 x 10⁻⁸</td>
</tr>
<tr>
<td>HPO₄²⁻</td>
<td>2.1 x 10⁻¹³</td>
</tr>
<tr>
<td>HC₂H₃O₂</td>
<td>1.8 x 10⁻⁵</td>
</tr>
<tr>
<td>NH₄⁺</td>
<td>5.6 x 10⁻¹⁰</td>
</tr>
</tbody>
</table>

PART A – PREPARATION OF A pH = 9.50 BUFFER SOLUTION – DEMONSTRATION

3. Your instructor will prepare a pH = 9.50 buffer by mixing 50.0 mL of a weak acid solution with the correct volume of a strong base solution. Reagents available to prepare each of the buffers in Parts A, B, and C found in Table 1.

NOTE: Use the $K_a$ values in Table 2 to choose which weak acid/conjugate base pair will be best suited for the assigned pH. Record the concentration of the stock weak acid and strong base solutions that will be used to prepare the pH = 9.50 buffer.

4. In calculation box 1, calculate the moles of the weak acid in the buffer to be prepared. Then in calculation box 2, using the Henderson-Hasselbach equation, the pH of the buffer, and the $pK_a$ of the weak acid, solve for the moles of strong base needed to prepare this buffer. Finally, in calculation box 3 calculate the volume of the strong base solution needed to prepare this buffer.

5. Open Logger Pro if it is not already open. Go to the Experiment menu and under Calibrate choose the channel with the pH probe (ex: “CH1:pH”). In the window that appears make sure the Calibration tab is chosen. Click on Calibrate Now. Rinse the pH meter with copious amounts of deionized water. Carefully blot dry. Place the pH meter in the pH 7 standard solution. Observe the voltage reading, found in the middle of the new window that opened up, and wait for the voltage reading to stabilize. In the field beneath Enter Value enter the pH value of the solution (7.00, not the voltage reading!) and click [Keep]. Rinse the pH meter with copious amounts of deionized water. Carefully blot dry. Place the pH meter in the pH 10 standard solution and wait for the voltage reading to stabilize. In the field beneath Enter Value enter the pH value of the solution (10.00, not the voltage reading!) and click [Keep]. When finished with this step click Done to close the window.
6. When not in use, place the pH sensor back into its aqueous potassium chloride storage solution.

7. Rinse the pH electrode thoroughly with deionized water and gently pat it dry before using it to measure the pH of a solution. Use only deionized water for rinsing the pH electrodes. Do not use the pH probe to stir solutions. Collect all rinsing’s in your waste beaker, which can be disposed of down the sink at the end of the laboratory period.

8. Place the pH probe into the solution. Stir the buffer solution, and when it appears the pH has stabilized, record the pH of the buffer solution in your Data Table.

**PART B – PREPARATION OF A pH = 5.00 BUFFER SOLUTION**

9. You will prepare a pH = 5.00 buffer by mixing 75.0-mL of a strong acid with the correct mass of solid sodium acetate trihydrate. Record the concentration of the stock strong acid that will be used to prepare the pH = 5.00 buffer.

**NOTE:** Look up the $K_a$ value in Table 2 for the weak acid you will be creating in your buffer solution.

10. In calculation box 4, calculate the moles of the strong acid that will be used in the buffer to be prepared. Then in calculation box 5, using the Henderson-Hasselbach equation, the pH of the buffer, and the $pK_a$ of the weak acid, solve for the moles of the sodium acetate needed to prepare this buffer. Finally, in calculation box 6, solve for the mass of the sodium acetate trihydrate needed to prepare this buffer. Have the instructor approve your calculations.

11. Prepare the buffer solution in a 250-mL beaker. When weighing out a solid reagent on the milligram balance, use the following steps:

(a) Transfer a small amount of the solid reagent from its reagent bottle to either a weighing cup or to a glass or porcelain container by pouring.

**CAUTION:** Never place your microspatula or scoopula into a reagent bottle.

**CAUTION:** Never pour solid or liquid reagents back into stock bottles. Any excess chemicals can be given to another student or properly discarded under the direction of the instructor.

(b) Place the clean, dry 250-mL beaker on a milligram balance pan. “Tare” the beaker by pressing one of the keys with the symbol “$\rightarrow T/0 \leftarrow$”. This will zero out the mass of the beaker.

(c) Remove the beaker from the balance chamber, add the chemical to the beaker

**CAUTION:** Never transfer chemicals to a container that is on ther pan in the balance chamber – remove the container from the balance chamber, add the chemical, and then return the container to the pan in the balance chamber.

(d) Place the beaker back on the balance pan, read the mass, and record it in your Data Table.

**NOTE:** If any chemical is spilled on the balance or on the lab bench, clean it up immediately, and dispose of it under the direction of the instructor. If there is any chemical left on the balance or the lab bench at the end of the lab period, the instructor will deduct one point from everyone’s lab score as a charge for cleaning up after you.

Record the mass of sodium acetate trihydrate in your Data Table.
12. If the laptop computer is turned on, proceed to step 13. If the computer is off, turn it on, and when the Log On dialogue box appears, for User Name: type in your Saddleback (or IVC) email address, and for Password: type in your Saddleback (or IVC) password. If any other dialogue boxes appears, select No or Cancel or Close.

13. Open Logger Pro if it is not already open. Go to the Experiment menu and under Calibrate choose the channel with the pH probe (ex: “CH1:pH”). In the window that appears make sure the Calibration tab is chosen. Click on Calibrate Now. Rinse the pH meter with copious amounts of deionized water. Carefully blot dry. Place the pH meter in the pH 4 standard solution. Observe the voltage reading, found in the middle of the new window that opened up, and wait for the voltage reading to stabilize. In the field beneath Enter Value enter the pH value of the solution (4.00) and click @Keep. Rinse the pH meter with copious amounts of deionized water. Carefully blot dry. Place the pH meter in the pH 7 standard solution and wait for the voltage reading to stabilize. In the field beneath Enter Value enter the pH value of the solution (7.00) and click @Keep. When finished with this step click Done to close the window.

14. When not in use, place the pH sensor back into its aqueous potassium chloride storage solution.

15. Insert the pH sensor into the buffer solution and click. In the lower-left hand corner of the Logger Pro window, you will see the pH in real time. Stir the buffer solution, and when it appears the pH has stabilized, record the pH of the buffer solution in your Data Table. We will see how close it is to pH = 5.00! The buffer solution can be washed down the sink with water when you are done.

PART C – BUFFER CAPACITY AND RANGE

16. On Logger Pro, click the Data Collection button (the one with a clock on it). You will see a Dialog Box. Change Mode from Time Based to Events with Entry. Type Drops for column name and Drops for short name. Change Units to Drops then click Done.

17. Obtain two different 25-mL portions of the 0.1 M Buffer Solution (0.1 M Na₂HPO₄ and 0.1 M NaH₂PO₄) and place them into two different 50-mL beakers.

18. To the first 25 mL portion, insert the pH sensor and click Collect. In the lower-left hand corner of the Logger Pro window, you will see the pH in real time. Stir the buffer solution, and when it appears the pH has stabilized, click @Keep. A new window will appear asking you to enter the number of drops added up to this point. Enter 0 into this window. This first data point (0 drops and corresponding pH) should now be recorded on the data columns at the left of the screen, and a red dot should show up on the graph indicating this data point.

19. Add 2 drops of 1.000 M HCl to the first portion of the buffer solution and stir. When it appears the pH has stabilized, click @Keep. Enter in the total number of drops that have been added (at this point, you have added 2). Repeat this step until 10 drops have been added. When you are done, click Stop. This portion of the buffer solution can be washed down the sink with water.

20. Rinse the pH meter with copious amounts of deionized water. Carefully blot dry. To the second 25 mL portion, insert the pH sensor and click Collect. A dialog box will appear. Click store latest run. In the lower-left hand corner of the Logger Pro window, you will see the pH in real time. Stir the buffer solution, and when it appears the pH has stabilized click @Keep. A new window will appear asking you to enter the number of drops added up to this point. Enter 0 into this window. This first data point (0 drops and corresponding pH) should now be recorded on the Data Table at the left of the screen, and a red dot should show up on the graph indicating this data point.
21. Add two drops of 1.000 M NaOH to the second portion of the buffer solution and stir. When it appears the pH has stabilized, click [Keep]. Enter in the total number of drops that have been added (at this point, you have added 2). Repeat this step until 10 drops have been added. When you are done, click [Stop]. This portion of the buffer solution can be *washed down the sink with water*.

22. Measure 10.0 mL of 0.1 M Buffer Solution (0.1 M Na₂HPO₄ and 0.1 M NaH₂PO₄) in a 100-mL graduated cylinder. Dilute the 10.0 mL with deionized water to a final volume of 100.0 mL, and stir. This produces a 0.01 M Buffer Solution (0.01 M Na₂HPO₄ and 0.01 M NaH₂PO₄).

23. Measure two different 25-mL portions of the 0.01 M Buffer Solution into two different 50-mL beakers. Repeat steps 16-19. When you click [Collect] a dialog box will appear. Click *store latest run*. Once you are done, you will have a plot with 4 curves.

24. Click on the *Autoscale Graph* button [A] (the one with the A on it) to enlarge the graph. Label each of the four curves by going to the *Insert* menu, selecting *Text Annotation*, and identifying the four curves as *0.1 M Buffer with HCl*, *0.1 M Buffer with NaOH*, *0.01 M Buffer with HCl*, and *0.01 M Buffer with NaOH*.

25. In Logger Pro, from the *File* menu, select *Print*, click *Print Footer*, type your name, and click *OK*. The selected printer should be *ISCI321000A*. Click *OK* to print, and your graph will be sent to the printer in the lab room and printed there. Retrieve your graph and attach it to your lab report.

26. Clean and wipe dry your laboratory work area and all apparatus. When you have completed your lab report have the instructor inspect your working area. Once your working area has been checked your lab report can then be turned in to the instructor.
### DATA TABLE

#### PART A

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration of Stock Weak Acid</td>
<td>.</td>
<td>M</td>
</tr>
<tr>
<td>Volume of Stock Weak Acid</td>
<td>.</td>
<td>mL</td>
</tr>
<tr>
<td>1 Quantity of Stock Weak Acid</td>
<td>.</td>
<td>mol</td>
</tr>
<tr>
<td>2 Quantity of Stock Strong Base</td>
<td>.</td>
<td>mol</td>
</tr>
<tr>
<td>Concentration of Stock Strong Base</td>
<td>.</td>
<td>M</td>
</tr>
<tr>
<td>3 Volume of Stock Strong Base</td>
<td>.</td>
<td>mL</td>
</tr>
<tr>
<td>Measured pH of the 9.50 pH Buffer</td>
<td>.</td>
<td></td>
</tr>
</tbody>
</table>

#### PART B

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration of Stock Strong Acid</td>
<td>.</td>
<td>M</td>
</tr>
<tr>
<td>Volume of Stock Strong Acid</td>
<td>.</td>
<td>mL</td>
</tr>
<tr>
<td>4 Quantity of Stock Strong Acid</td>
<td>.</td>
<td>mol</td>
</tr>
<tr>
<td>5 Quantity of Sodium Acetate Trihydrate</td>
<td>.</td>
<td>mol</td>
</tr>
<tr>
<td>6 Calculated Mass of Sodium Acetate Trihydrate</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>Measured Mass of Sodium Acetate Trihydrate</td>
<td>.</td>
<td>g</td>
</tr>
<tr>
<td>Measured pH of the 5.00 pH Buffer</td>
<td>.</td>
<td></td>
</tr>
</tbody>
</table>
## CALCULATIONS

1. 

2. 

3.
QUESTIONS

1. Based upon the four curves from Part C, what can you conclude about the effect of concentration on a buffer’s effectiveness?

2. Identify the weak acid from Table 2 that should be used to prepare a pH = 2.00 buffer.

3. A pH = 2.00 buffer can be prepared by mixing 75.0 mL of 0.40 M NaH₂PO₄ with the correct volume of 0.1000 M HCl. Determine the volume of the 0.1000 M HCl needed.