Experiment 5 – pH Titration of Acetic Acid with Sodium Carbonate

Materials Needed to Complete This Experiment (https://youtu.be/EVXdrGqyB9E) Note to Student: Please review this video before you begin the lab.

- Na_2CO_3 solution from Experiment 4
- Calibrated plastic pipette from Experiment 2
- 50 mL beaker, 100 mL beaker, 250 mL beaker, 500 mL beaker, waste beaker
- pH tester including calibration materials
- Distilled white vinegar
- Distilled / deionized water
- Goggles

Common Lab Procedures/Skills in This Lab (new skills in bold)

1. Calibrate a pH tester (https://youtu.be/9jfzuruainm)
2. Condition* pipette and beakers
3. Measure and record the pH
4. pH titration (https://youtu.be/fjeve-pvoe8)
5. Deliver 2.0 ml using the pipette*
6. Calculate concentration of unknown acid (https://youtu.be/jjqojfq1-m8)
7. Graphing using excel*
8. Graphically determine the equivalence point (https://youtu.be/esydc1d5pdc)
9. Conduct a q-test on a potential outlier (https://youtu.be/rosc2ju702u)

Introduction

Objectives:

- Plot a pH Titration Curve for a weak acid/weak base titration.
- Compare this curve to a similar curve for a strong acid/strong base titration.
- Determine the concentration of an unknown acid, the concentration of acetic acid in vinegar.
- Compile a data set of values for you and two other classmates.
- Within that data set, determine if there is an outlier that can be discarded using a q test.

In this experiment you will explore acid-base chemistry. You will perform a weak-acid/weak-base titration using acetic acid and sodium carbonate. The titration reaction in this lab is:

$$CH_3COOH_{(aq)} + Na_2CO_{3(aq)} \rightarrow NaHCO_{3(aq)} + NaCH_3COO_{(aq)}$$

A titration is a technique where a solution of a known concentration is used to determine the concentration of an unknown solution. Typically, the titrant (known solution) is added from a buret to a known quantity of the unknown solution until the reaction is complete. Knowing the volume of the titrant added allows the determination of the unknown concentration. Often, an indicator is used to signal the end of the reaction, known as the endpoint.

In this experiment, the calibrated 3 mL pipette from experiment 2 will be used in lieu of the buret to keep track of known volumes and the pH tester will be used instead of an indicator to determine the equivalence point. The total volume and pH will be used to plot a titration curve.

The equivalence point is slightly different from the endpoint of a titration. The endpoint is when the indicator changes color. This does not always correspond to the equivalence point. The equivalence point is the point in a titration when the amount of added titrant is chemically equivalent to the amount of analyte in the sample.

A titration curve is a plot of the pH of an acid versus the volume of base added (or vice versa). The titration curve gives a good description of how an acid-base reaction progresses. The pH will start out low then will increase as it reaches the equivalence point, where the moles of the acid equals the moles of the base according to the reaction stoichiometry. As the solution becomes more basic, it will slowly rise and level off as an excess amount of base is added.

There are several types of acid-base titrations; each with a characteristic curve. For strong acid-strong base titrations, there is a very sharp change in pH over the addition of less than a drop of base. The equivalence point lies at the midpoint of the vertical portion of the curve. The pH changes quickly near the equivalence point and the pH = 7.0 (Figure 1a). In other types of titrations, the pH does not equal 7 as can be seen in a strong acid-weak base titration (Figure 1b). The strong acid-weak base also has a sharp change in pH near the equivalence point.

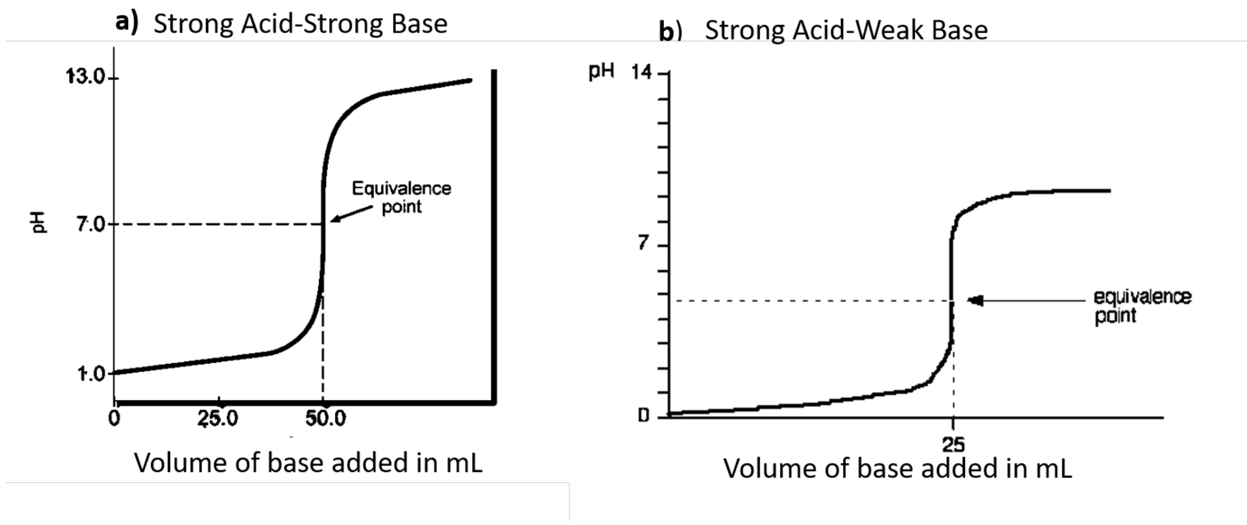

Figure 1. a) Strong Acid-Strong Base titration curve b) Strong Acid-Weak Base titration curve

As noted earlier, a weak acid-weak base titration is the focus of this experiment. Figure 2 shows an example of a weak acid-weak base titration curve. Notice that the curve lacks a sharp pH change. In addition, the equivalence point for weak acid-weak bases may not be exactly at pH = 7.00.

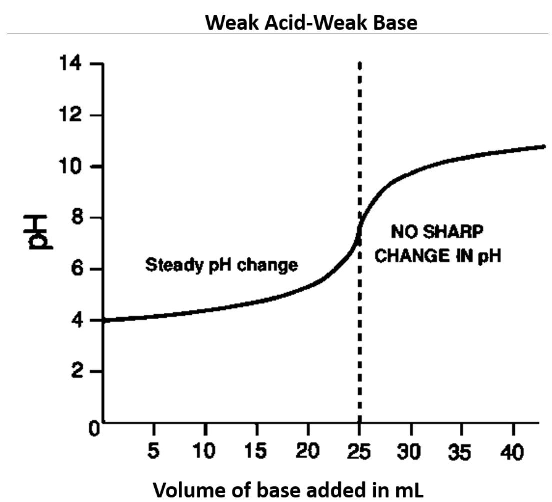

Figure 2 - Weak Acid-Weak Base titration curve

Following the completion of the experiment, you will perform a Q-test. The Q test is a test to see if we can assume with greater than 90% statistical confidence that one point, called the "outlier", is in error and can be thrown out. The following table below is $Q_{critical}$ tabulated with varying number of observations. For this lab, you will likely have 9 values in your data set: $Q_{critical}$ = 0.44. Section B in the calculations section describes how to conduct a Q-test on a potential outlier.

Number of Observations	$Q_{Critical}$
3	0.94
4	0.76
5	0.64
6	0.56
7	0.51
8	0.47
9	0.44
10	0.41

Prelab questions

1. In Experiment 2, you calibrated a pipette. Note the calibrations for 2 mL, 1 mL, 0,5 mL, and one drop.

   For 2 mL, I delivered ________ mL. For 1 mL, I delivered ________ mL.

   For 0.5 mL, I delivered ________ mL. For 1 drop, I delivered ________ mL.

2. In Experiment 4, you prepared a solution of Na_2CO_3. What is the concentration of that solution?

   The concentration of the Na_2CO_3 is ___________ M.

Procedure – You will work on this lab independently (no lab partners).

A. **CALIBRATE THE pH TESTER:** Follow the directions for calibrating your pH tester using the pH Buffer Powder Packets included with the tester. (**Video 1 Procedure/Skill 1 above**)

Here is one version of the directions:

1. Dissolve the packet labeled pH 6.86 in 250 mL of distilled water in a clean, dry beaker. If the beaker is not dry, **CONDITION** the beaker with distilled water before making the solution.

2. Dissolve the packet labeled pH 4.01 in 250 mL of distilled water in a different clean, dry beaker. If the beaker is not dry, **CONDITION** the beaker with distilled water before making the solution.

   The following is based on the instructions that came with the pH tester:

3. Turn on the pH tester.

4. **MEASURE THE pH:**
   a. Remove the cap.
   b. Rinse the pH electrode (glass bulb inside plastic casing) with distilled water. Gently shake off any excess water.
   c. Immerse the electrode into the pH 6.86 solution up to the area that was covered by the cap.
   d. Stir for 5-10 seconds.
   e. Stop stirring and wait for the reading to stabilize.

5. Press the “CAL” key for 2 seconds and release. The pH tester can automatically identify the buffer solution. The display will start flashing “6.86”. Wait until the display stops flashing and displays “end”.

6. Rinse the electrode with distilled water.

7. Repeat steps 4-7 with the pH 4.01 solution.

8. Once the pH tester is calibrated, it will be calibrated for the rest of the experiments this semester.

9. If there is a problem with the calibration, note what happened in your data section and let your instructor know. There are some trouble shooting notes that are part of the instructions that came with the pH tester.

B. **pH TITRATION** (**Video 2 Procedure/Skill 4 above**)

1. Pour ≈150 mL of distilled white vinegar into a clean and dry 250 mL beaker. If the beaker is not dry, then **CONDITION** the beaker with the vinegar and then add the vinegar.

   Note: conditioning the beaker with vinegar makes sure that the vinegar is not diluted when added to the beaker.

2. Pour ≈40 mL of the Na_2CO_3 solution from Experiment 4 into a clean, dry 50 mL beaker. If the beaker is not dry, then **CONDITION** the beaker with the Na_2CO_3 solution and then add the Na_2CO_3 solution.

   Note: conditioning the beaker with Na_2CO_3 solution makes sure that the Na_2CO_3 solution is not diluted when added to the beaker.

3. **CONDITION THE PIPETTE** with vinegar**. CONDITION** or rinse the 100 mL beaker with distilled water.

   Note: you will use the 100 mL beaker for your titration. For titrations, the only thing that is important is the moles of either acid or base- not their concentrations, that is why this beaker is conditioned or rinsed with distilled water.

4. **DELIVER** 20.00 mL of vinegar **USING THE PIPETTE** to deliver 2.00 mL ten times into the 100 mL beaker.

5. **MEASURE AND RECORD THE pH:**
   a. Remove the cap.
   b. Rinse the pH electrode (glass bulb inside plastic casing) with distilled water. Gently shake off any excess water.
   c. Immerse the electrode into the solution up to the area that was covered by the cap.
   d. Stir for 5-10 seconds.
   e. Stop stirring and wait for the reading to stabilize.
   f. Record the pH.
   g. Rinse the pH electrode with distilled water. Gently shake off any excess water. Set the pH tester aside.

6. **CONDITION THE PIPETTE** with distilled water followed by Na_2CO_3 solution**.**

7. **DELIVER** 2 mL of Na_2CO_3 solution using the pipette into the vinegar. **MEASURE AND RECORD THE pH.**

8. Repeat step 7 three times.

9. Each time you add the Na_2CO_3 solution to the vinegar, the pH changes by a certain amount. As you get closer to the equivalence point, you will notice that bigger changes in pH occur for the same addition of Na_2CO_3 solution. This is your signal that you should start adding smaller volumes of Na_2CO_3 solution each time. Between pH of 5 and 6, add 1 mL at a time. Between pH of 6 and 8.5, add 0.5 mL. Above pH of 8.5, add 2 mL until, you reach a pH of 9.5 or higher. For this titration, you don't have to get down to adding drops.

10. Repeat steps 3-9 two more times to get two more pH titration curves. Don't forget to **CONDITION** your pipette every time you change solutions. You may have to refill the 50 mL beaker with Na_2CO_3 solution. You will have to make a new data table for each new titration.

Calculations

A. **CALCULATE CONCENTRATION OF UNKNOWN ACID**

1. Complete Table 1.

2. **GRAPHING USING EXCEL:** Graph each set of data in Excel with Total mL added with pipette on the x-axis and pH on the y-axis. Make sure each graph has a title, axes titles with units, and that your axes limits have your data take up most of the graph (usually, Excel does this for you automatically).

3. **GRAPHICALLY DETERMINE THE EQUIVALENCE POINT:** The equivalence point is the point on the graph when the slope is the steepest. The slope is $\Delta y/\Delta x$. Find the two data points with the steepest slope. Take the average of the number of mL of Na_2CO_3 solution for these two points. This average mL is the mL of Na_2CO_3 solution at the equivalence point. Do this for each of your three graphs. **(Video 4 Procedure/Skill 8 above)**

   Note: for weak acid/weak base titrations (like this one), the equivalence point may not be exactly at pH = 7.00. In fact, you will likely find the equivalence point to be at a pH that is a little higher than pH = 7.00.

4. Use the volume of Na_2CO_3 solution, M Na_2CO_3 solution, and the volume of vinegar to solve for the concentration of acetic acid in vinegar. Do this for each of your three trials. **(Video 3 Procedure/Skill 6 above).**

5. Calculate the average M of acetic acid

6. Calculate the standard deviation

B. Check for an outlier in a data set

1. Get the values for the concentration of acetic acid in vinegar from two classmates. This should give you 9 values. List the names of the students with their values.

2. **CONDUCT A Q-TEST ON A POTENTIAL OUTLIER: (Video 5 Procedure/Skill 9 above)**

a. The potential outlier, or point that we are considering if we can ignore it, is the point in your data set with the largest gap.
b. Of the nine values, circle the highest and lowest values.
c. For the highest and lowest values, calculate the "gap":

$$\text{gap} = |\text{potential outlier} - \text{next closest point}|$$

d. Calculate the range: range = highest – lowest
e. Calculate the parameter Q_{data}:

$$Q_{data} = \frac{\text{gap}}{\text{range}}$$

f. Find the value of $Q_{critical}$ (given in table form in the introduction).
g. If $Q_{data} > Q_{critical}$, then the potential outlier is an outlier and can be ignored from the data set.

3. Calculate the average M of acetic acid

4. Calculate the standard deviation.

Post Lab Questions

1. Draw by hand on one of your Excel graphs what a strong acid/strong base titration curve would look like.

2. Calculate the molarity of a solution of potassium hydroxide if 19.55 mL is required for the titration of a 20.20 mL sample of a 0.3467 M sulfuric acid solution.

Results:

Table 1

Data: First Titration		Plot these columns	
Nominal mL added by pipette	Actual mL added with pipette	Total mL added with pipette	pH
0 mL	0 mL		
+ 2 mL			
+ 2 mL			
+ 2 mL			
+ 2 mL			

Etc., for as many points as you have for this data set.

Table 2

Summary Table	
Average concentration and standard deviation of acetic acid Part A in calculations	
Average concentration and standard deviation of acetic acid Part B in calculations	