Experiment 30: Applying the Concept of Buffers

(Part B of this experiment was adapted from Colby College, CH 142, Spring 2012.)

Purpose The purpose of this experiment is to determine the Ka of a weak acid; acetic acid. In order to do this, the acid solution will be titrated with an NaOH solution, using two different methods of titration. (The NaOH solution will first need to be standardized to determine its exact concentration.)

Background

The NaOH solution is approximately 0.075 M, however, the exact concentration is not known. Therefore, the NaOH solution will have to be standardized. That means you will use pure potassium hydrogen phthalate (KHP) as the acid in a titration with this NaOH solution. The moles of KHP will be known and the volume of NaOH used will be known (final – initial buret volumes). You will be calculating the molarity concentration of NaOH (M = moles / Liters). The NaOH and KHP react on a 1:1 basis as follows

$$KHP_{(aq)} + NaOH_{(aq)} \rightarrow KNaP_{(aq)} + H_2O_{(l)}$$

After the NaOH solution is standardized, you will use it to titrate the acid solutions. The Ka values will be determined with two different methods. The concentration of acetic acid and sulfuric acid will be also be determined.

Acetic Acid:

Titration method 1: The monoprotic acid will be titrated twice with the standardized NaOH solution. (This lets you calculate the concentration.) You will use the titrated solution to make a buffer solution. The buffer will be prepared by mixing the titrated solution with an equal volume of ***prepared*** acid solution.

When the acetic acid is titrated, the acid is used and its conjugate weak base is made. At the equivalence point, the titrated solution contains the weak conjugate base and spectator ions. When a portion of original weak acid (diluted to match the total volume of the titrated solution) is added to the titrated solution, a buffer solution is created that contains [HA] = [A^-]. When the concentrations of HA and A^- are equal, the pH equals the pKa. You will create this buffered solution and then measure the pH. The pH value will be used to calculate the Ka value. (pH = pKa pKa = 10^{-pKa})

Titration method 2: This acetic acid will be titrated with the NaOH solution. The pH of the acid solution will be measured during the titration, after each increment of NaOH is added. An S-shaped titration curve will be made with Excel or Google Sheets so that the equivalence point and Ka can be determined.

Chemicals

Pure potassium hydrogen phthalate (KHP), FW 204.2 g/mole	NaOH solution, approximately 0.08 M
Acetic acid solution, 0.050 M	
Phenolphthalein solution, 1%	
Commercial pH buffers to check the pH meter (pH = 4.00, pH = 7.00)	

Equipment

Erlenmeyer flasks, 125 mL	Beaker, 100 mL	Beakers, assorted sizes
Pipet, 20 mL, 10 mL	Buret, buret stand & clamp	Lab balance
pH meter		

Procedure

Part A: Standardization of the NaOH Solution

1) Use the lab balance and tared weigh paper to weigh out 0.230 g – 0.250 g of KHP. Record the exact mass and then transfer it into a 125 mL Erlenmeyer flask.
2) Add 20 mL of DI water to the flask to dissolve the KHP. Use a glass stir rod to break up any chunks of KHP, stir to dissolve, and mix well.
3) Add three drops of phenolphthalein indicator to the KHP solution in the flask; swirl to mix.
4) Set up the buret as indicated in the titration video in Blackboard:
    a. obtain the buret, buret stand and buret clamp
    b. rinse the buret with DI water
    c. rinse the buret with three, 5 mL portions of the NaOH solution
    d. fill the buret with the NaOH solution
    e. drain some of the NaOH to remove the air bubble in the buret tip
5) Record the initial volume of NaOH in the buret. All volume readings must have two digits after the decimal point, even if one or both digits are zero.
6) Place the Erlenmeyer flask with the KHP solution under the buret tip.
7) Titrate with the NaOH until the solution becomes a faint pink color. The color must linger for at least one minute. If the solution color is dark pink, you must discard the trial and do another trial.
8) Repeat steps 1 – 7 (omit step 4, the buret is already setup) until you have two usable titrations. Each trial must be done from start to finish before beginning the next trial.
9) Calculate the molarity of NaOH for each trial, and then calculate the average M_{NaOH}. You will use this average molarity for your calculations in parts B and C.

Part B: Titration of the Acetic Acid Solution with titration method 1

1) Pipet 20.0 mL of acetic acid solution into an Erlenmeyer flask.
2) Add three drops of phenolphthalein indicator into the acid; swirl to mix.
3) Refill the buret with NaOH solution if necessary.
4) Record the initial buret volume (2 digits after the decimal point).
5) Titrate the acid solution until the faint pink endpoint is reached.
6) Record the final buret volume.
7) Measure the total volume of solution in the flask with a clean, dry graduated cylinder. Record this volume in your notebook.
8) Transfer this solution back to the Erlenmeyer flask and <u>SAVE THIS SOLUTION FOR LATER</u>
9) Pipet 20.0 mL of acetic acid solution into the relatively dry graduated cylinder.
10) Add enough DI water to the cylinder to make the total volume equal to the total volume of titrated solution found in step 7. This new solution is your <u>*prepared*</u> acid solution.
11) Combine the <u>*prepared*</u> acid solution with the titrated solution in the Erlenmeyer flask. Mix well. This is your trial 1 buffered solution of acetic acid / acetate.
12) Measure the pH of this buffered solution. This pH is equal to the pKa of acetic acid.
13) Repeat steps 1 – 12 for trial 2.

Part C: S-Shaped Titration Curve with titration method 2

1) Pipet 20.0 mL of acetic acid solution into a clean, dry 100 mL beaker.
2) Add three drops of phenolphthalein indicator into the acid; swirl to mix.
3) Refill the buret with NaOH solution if necessary (you will need approx. 35 mL).
4) Record the initial buret volume. Start at 0.00 mL. (2 digits after the decimal point).
5) Check the pH meter with the pH = 4.00 buffer solution.
6) Put the pH electrode in the 100 mL beaker and record the pH of the acetic acid solution.
7) Start to titrate the acid solution, adding 2 mL of NaOH solution from the buret as each increment of base. After each increment, swirl to mix well and record the pH of the solution in the beaker. The pH electrode should be sitting in the beaker during the titration, off to the side so the stream of NaOH solution from the buret does not hit the electrode. Do 5 increments of NaOH addition.
8) Continue to add NaOH by 1 mL increments, mixing and measuring the pH after each increment. Do this for 3 increments.
9) Continue to add NaOH by 0.5 mL increments, mixing and measuring the pH after each increment. Do this until the pH jumps up significantly; pH of approximately 3.40 to pH of approximately 11.
10) Continue to add NaOH by 1 mL increments, mixing and measuring the pH after each increment. Do this for 2 increments.

At this point, you have enough data to plot your S-shaped titration curve. Tabulate the total volume of NaOH solution added to the beaker at each increment and the pH of the solution in the beaker. There is a video in Blackboard to show you how to make the S-shaped titration curve.

For the Calculations / Results section of your notebook:

Part A

1) For each trial of titrated KHP, calculate the molarity of the NaOH solution.
2) Calculate the average molarity of NaOH ((T1 + T2) / 2). Use this average molarity of NaOH for the calculations in Part B and Part C.

Part B

3) For each trial of titrated acetic acid, calculate the molarity of the acid.
4) Calculate the average molarity of acetic acid ((T1 + T2) / 2).
5) Use the pH of each buffered solution of acetic acid/acetate to calculate the Ka.
6) Calculate the average the Ka value ((T1 + T2) / 2).

Part C

7) Plot an S-shaped titration curve that shows the equivalence point for the titration of acetic acid.
8) On the graph, drop straight down from the center of the vertical pH jump to determine the volume of NaOH at the equivalence point. Divide the equivalence point volume by 2, and use this ½ volume to determine the pH that equals the pKa. Watch the video in Blackboard to see how to do this.
9) Calculate Ka for CH_3COOH. At the ½ equivalence point, pH = pKa, and Ka = 10^{-pKa}.
10) Calculate the molarity of the acetic acid solution originally used with the equivalence point NaOH volume obtained from the graph. Remember, the stoichiometry for this titration is 1 NaOH : 1 CH_3COOH