CH 223 Spring 2024: **"Acid & Base Titrations** *(in class)"* ***Lab*** *- Instructions*

Note: **This is the lab for section 01 and H1 of CH 223 only.**

- *If you are taking section W1 of CH 223*, please use this link: http://mhchem.org/q/5b.htm

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Step One:

Get a printed copy of this lab! You will need a printed (hard copy) version of pages Ia-5-2 through Ia-5-9 to complete this lab. If you do not turn in a printed copy of the lab, there will be a 2-point deduction.

Step Two:

Bring the printed copy of the lab with you on Monday, April 22 (section 01) *or* **Wednesday, April 24 (section H1.)** During lab in room AC 2507, you will use these sheets (with the valuable instructions!) to gather data, all of which will be recorded in the printed pages below.

Step Three:

Complete the lab work and calculations on your own, then **turn it in** (pages Ia-5-5 through Ia-5-9 *only* to avoid a point penalty) **at the beginning of recitation to the instructor on Monday, April 29 (section 01)** *or* **Wednesday, May 1 (section H1.)** The graded lab will be returned to you the following week during recitation.

If you have any questions regarding this assignment, please email (mike.russell@mhcc.edu) the instructor! Good luck on this assignment!

Acid & Base Titrations

During the next several labs you will perform titrations on acids and bases to determine various properties about these solutions. This first lab is designed as an introduction to titration with acids and bases. The focus of this lab will be to familiarize you with the Vernier computer system and to perform a titration using it. The program you use can be modified as needed in the following lab classes.

A **titration** is a process by which a solution of known concentration is quantitatively added to a solution of unknown concentration in order to determine its concentration. You will be using burets to perform the titrations. The titration **end point** or **equivalence point** is when stoichiometrically equivalent amounts of the two substances are present. Therefore, it is necessary to create a method for determining the endpoint of the titration. In an acid-base titration, the change in the acidity or pH of the solution is a convenient method to determine this equivalence point.

There are two methods to measure the pH of a solution. In the first method a chemical called an **indicator** is used that changes color upon a change in the acidity of the solution. Litmus and phenolphthalein are examples of common indicators. The second method is to use a **pH meter** to measure the pH of the solution as the titration proceeds.

In this lab you will use a Vernier interface with a pH probe to measure the $[H_3O^+]$ of the solution. The program will graph the progress of the titration as the titrant is added from the buret (measured with a drop counter.) A plot of the pH of a solution against the volume of titrant added is called a **titration curve**. From the titration curve, the equivalence point can be determined as the point of maximum slope. For an acid-base titration, the equivalence point occurs when moles of acid equal moles of base: $[H_3O^+] = [OH^-]$. Furthermore, the equivalence point will reveal whether the solution consists of a strong or weak acid.

For an acid, HA, in solution, the equilibrium constant K_a for the process can be determined:

$$HA_{(aq)} + H_2O_{(l)} \rightleftharpoons H_3O^+_{(aq)} + A^-_{(aq)} \qquad K_a = \frac{[H_3O+][A^-]}{[HA]}$$

Recall that in solution, there is also a second equilibrium of concern:

$$\mathbf{2\,H_2O \rightleftharpoons H_3O^+ + OH^-, \qquad K_w = [H_3O^+][OH^-] = 1.00 * 10^{-14}}$$

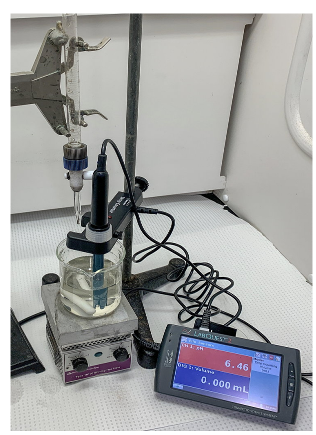
For a **strong acid**, K_a is so large that the acid dissociates completely into H_3O^+ and A^- such that $[HA] = [H_3O^+]$ even in the untitrated state. At equivalence, the dissociation of water governs the pH, and the pH = 7.

For a **weak acid**, K_a is small and hence influences the pH. The initial pH of the solution will appear higher than that of a strong acid and the pH at the equivalence point is not 7. To calculate K_a, you will need to determine the pH halfway to the equivalence point. At the halfway point or **half equivalence point**, half of the acid has been titrated such that $[A^-] = [HA]$. This will reduce the equilibrium expression to $K_a = [H_3O^+]$ or **pH = pK_a**. Therefore, if the volume at the equivalence point is determined, the pH at half that volume will reveal pK_a.

PERFORMING THE EXPERIMENT - *Instructions*

You will use a Vernier interface and equipment to titrate three acids using NaOH. Note the setup of the Vernier equipment carefully; you shall perform several similar titrations during this lab period and in the weeks to come. At the end, optionally upload your graphs (and maybe data files) to your email account and analyze them later at your convenience.

1. Obtain approximately 65 mL of NaOH solution in a 250 mL beaker.
2. Obtain a 25 or 50 mL buret and connect it to a ring stand using a buret clamp. Rinse the buret with a few mL of the NaOH and discard into a waste jar. Now fill the buret to the 0.00 mL line. Drain a small amount of NaOH solution into the 250 mL beaker so it fills the buret's tip. Use a disposable pipet to ensure the NaOH volume is still at 0.00 mL.
3. Connect the pH Sensor to CH 1 of the Vernier LabQuest 2 Interface. Lower the Drop Counter onto a ring stand and connect it to DIG 1. Choose **New** from the **File** menu.
4. ***Calibrate the Drop Counter*** so that a precise volume of titrant is recorded in units of milliliters.
   a. Choose **Sensors ► Calibrate ► Drop Counter** from the Sensors menu. *If you do not see the "Sensors" entry at the top, push the "Meter" icon in the upper left corner.*
   •*If you have previously calibrated the drop size* of your buret and want to continue with the same drop size, tap **Equation** after selecting the "Drop Counter" in the "Calibrate" menu. Enter the value for drops/mL. Select Done, then OK. Proceed directly to the next section.
   •If you want to perform a new calibration (i.e. if you change burets), continue with this step.
   b. Select **Calibrate Now**.
   c. Place a 10 mL graduated cylinder directly below the slot on the Drop Counter, lining it up with the tip of the buret.
   d. Open the valve on the buret. Slowly add NaOH from the buret at a slow rate (~1 drop every second). You should see the drops being counted on the screen.
   e. When the volume of NaOH solution added from the buret is between 3 and 9 mL, close the buret.
   f. Enter the precise Volume of NaOH to 0.01 mL. Select **Done**. **Record** the number of drops/mL displayed on the screen (**Sensors ► Calibrate ► Drop Counter** then **Equation**) for possible future use. Select OK.
   g. It's also recommended to **calibrate the pH meter (Sensors ► Calibrate ► pH Meter)**. Use a two point calibration (at pH of 4 and 10 using buffer solutions in small beakers.)
5. Discard the NaOH solution in the graduated cylinder and set it aside. Fill the NaOH in the buret to the 0.00 mL level.
6. Using a graduated cylinder, measure **5.00 mL HCl** (**record the concentration!**) into a 250 mL beaker. Add about 100 mL of water.
7. Assemble the apparatus.



   a. Place a magnetic stirrer under or near the ring stand with the buret. Place a stir bar in the 250 mL beaker with the HCl and place it on the magnetic stirrer.
   b. Rinse the pH sensor with water, then insert it through the large hole in the Drop Counter and attach it to the ring stand. Make sure the stir bar does not touch the bulb of the pH sensor.
   c. Adjust the positions of the Drop Counter and buret so they are both lined up. Test the positioning by releasing a few drops of NaOH; if the red light on the drop counter appears and disappears, all is well; if not, readjust the buret so the drops are counted. Also make sure the pH Sensor is just touching the bottom of the beaker.

8. Turn on the magnetic stirrer so that the stir bar is stirring at a fast rate, but not hitting the pH sensor bulb.

9. You are now ready to begin collecting data! Check to see that the pH value is **acidic** (you are starting with an acid.)

10. **Start data collection** by pressing the "start" button (the **green arrow** in the corner of the LabQuest.) No data will be collected until the first drop goes through the Drop Counter slot. Open the buret valve so that about 1 drop is released every 1 second or so. When the first drop passes through the Drop Counter slot, check the graph to see that the first data pair was recorded.

11. Continue watching your graph to see when a large increase in pH takes place—this will be the equivalence point of the reaction. When this jump in pH occurs, let the titration proceed for several more milliliters of titrant, then **stop data collection** (the **red square** in the corner) to view the graph of pH vs. volume. Make sure you see the "S" curve (or more if the acid is polyprotic.)

12. Dispose of the beaker contents in a waste jar, remembering not to put the stir bar in the waste jar.

13. **Examine the data** on the displayed graph of pH vs. volume (in mL) using your finger or the stylus to estimate the equivalence point volume and pH. **Record the pH value** in your notebook and **record the NaOH volume in mL** at the equivalence point. *Note:* if this is a weak acid graph, also record the half equivalence volume and the half equivalence pH value.

14. ***Optional:*** **Email copies** of each graph for later analysis (if this is available.) In the LabQuest app, select (upper left corner) **File -> Email -> Graph**, fill out the "To" line (and maybe the CC: line(s)), then select "Send." *Note:* **emailing a graph is optional, but nice to have *if* it is available.**

15. ***Optional*** *step for Excel power users:* You can make your own graph; to do so, **File -> Email -> Text File**, send it to yourself, then import into Excel (the data file is a Tab delimited text file) and create an xy-scatter graph.... if you try this optional step and get stuck, email the instructor. Do *not* email yourself the "Data File" unless you have the Vernier software installed on your device.

16. You are now done with Experiment #1 (HCl + NaOH). ☺

17. **Experiment #2** will be similar, but this time with **acetic acid**. Repeat the above procedure with HCl but substitute acetic acid for HCl (use the same volumes, etc.) Make sure to **rinse the pH meter with water before inserting it into a new solution.** Remember to record the acetic acid concentration!

18. **Experiment #3** will be similar, but this time with **phosphoric acid**. Repeat the above procedure with HCl but substitute phosphoric acid for HCl (use the same volumes, etc.) Remember to record the phosphoric acid concentration! Make sure your pH at the end of the titration if quite basic…. this is a polyprotic acid, and the graph should look quite different.

Use these instructions as you gather data and analyze three titrations.

Special note: we will be using these instructions during the next lab (Titration of Weak Acids), so bring these pages to that lab as well!

Acid & Base Titrations - *Worksheet*

Submitting graphs with this lab is optional - ask the instructor if this option is available.

YOUR NAME: ______________________ ***LAB PARTNER:*** ________________

Purpose: To explore acid-base pH titrations for three types of acid + strong base systems.

Experiment #1: HCl + NaOH

Determine the equivalence point volume and pH from the graph. Using the equivalence point volume and the exact concentration of the HCl, determine the **molarity of the pure** (initial) **NaOH solution** (i.e. before the NaOH reacted with the HCl.)

Equivalence point volume of NaOH (mL): ______________ Equivalence point pH: ______________

Concentration of HCl used in lab (M): ______________

Using the equivalence point volume and the exact concentration of the HCl, determine the **molarity of the pure** (initial) **NaOH solution** (i.e. before the NaOH reacted with the HCl.) *Show your work!*

HCl is a: strong acid strong base weak acid weak base *(Circle one)*

NaOH is a: strong acid strong base weak acid weak base *(Circle one)*

What *should* be the equivalence point pH be for this experiment?

less than 7 equal to 7 greater than 7 *(Circle one)*

Assume it takes 2.80 mL of 0.1100 M NaOH to titrate 5.00 mL of HCl. **Calculate the concentration of HCl using this data** (and show your work!)

<u>Experiment #2</u>: Acetic Acid - NaOH

Determine the equivalence point volume and pH from the graph. Based on the volume added at the equivalence point, **record the pH and volume at the half-equivalence point**. **Determine the K_a value based on the half equivalence point data**.

Equivalence point volume of NaOH (mL): ______________ Equivalence point pH: ______________

Half Equivalence point volume of NaOH (mL): __________ Half Equivalence point pH: __________

Experimental Ka of acetic acid: ______________________________ *Show work*

acetic acid is a: strong acid strong base weak acid weak base *(Circle one)*

What *should* be the equivalence point pH be for this experiment?

less than 7 equal to 7 greater than 7 *(Circle one)*

Why is the half-equivalence pH (and half-equivalence volume) important in this titration? Explain.

Acetic acid has a known K_a of 1.8×10^{-5} via the literature. Find the **percent error** for this experiment.
Recall: **Percent error = absolute value{(actual - experimental)/ actual}*100%**

Draw the Lewis structure of acetic acid showing all bonds and lone pair electrons:

Experiment #3: **Phosphoric Acid - NaOH**

Record the volume *and* pH values at the TWO equivalence points (first *and* second) for the phosphoric acid solution. (In most cases, you will not be able to observe the third equivalence point.) Use your data and/or graphs to determine the exact equivalence points.

Record the pH and volume at *each* half-equivalence point (first *and* second)**. Determine the value of K_{a1} and K_{a2} using the two half equivalence point values** on your graph. Note that to find K_{a2}, take the mid-point volume between the first equivalence point and the second equivalence point.

phosphoric acid is a: strong acid strong base weak acid weak base *(Circle one)*

From the graph, estimate the **first** equivalence volume: ________________ mL

From the graph, estimate the **first** equivalence pH: ________________

From the graph, estimate the **first** half-equivalence volume: ________________ mL

From the graph, estimate the **first** half-equivalence pH: ________________

Using this value, calculate the value of K_{a1} for phosphoric acid: ________________ *Show work*

Write the balanced equation used for K_{a1} of phosphoric acid

$H_3PO_4(aq) + H_2O(l) \rightleftharpoons$ ___________ + ____________________

Experiment #3: Phosphoric Acid - NaOH *Continued*

From the graph, estimate the **second** equivalence volume: ________________ mL

From the graph, estimate the **second** equivalence pH: ________________

From the graph, estimate the **second** half-equivalence volume: ________________ mL

From the graph, estimate the second half-equivalence pH: ________________

Using this value, calculate the value of K_{a2} for phosphoric acid: ________________ *Show work*

Write the balanced equation used for K_{a2} of phosphoric acid:

_____________ + **$H_2O(l)$** $\rightleftharpoons$ ______________ + ____________________

Postlab Questions:

1. If you titrate 100 mL of an unknown strong acid solution with 0.1 M NaOH, will the pH ever reach a value of 13? Explain.

2. What should the pH value be for the third half-equivalence point for phosphoric acid? Explain. Calculate what the exact value should be by using the table of acid dissociation constants in problem set #2.

3. What is the molarity of an HCl solution if 25.00 mL of the acid solution required 42.68 mL of 0.2525 M NaOH solution to reach the equivalence point?

4. A student titrates 50.0 mL of a weak acid, HA, with 0.100 M NaOH. It requires 43.68 mL of 0.100 M NaOH to reach the equivalence point of the titration.

    a. Calculate the moles of HA present.
    b. Calculate the original (undiluted) concentration of the weak acid solution.
    c. It took 21.84 mL of 0.100 M NaOH to reach the half-equivalence point for this reaction, and the pH of the solution at this point was 6.00. Calculate the K_a for the weak acid.

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