

Name: \_\_\_\_\_

## Ka of Unknown Acid Pre-Lab

1. Use the following data from a titration calculate what mass of acid would be needed to completely react with **9.50 ml of titrant**, if the molarity of base was 0.10 M of NaOH.

Sample Mass 0.30g

Initial reading 0.10 ml

Initial reading 0.00 ml

Final reading 25.00 ml

Final reading 3.00 ml

Volume of Titrant \_\_\_\_\_ ml

Volume of Titrant \_\_\_\_\_ ml

Answer: \_\_\_\_\_

2. If 0.537 grams of HA (an unknown monoprotic acid) was titrated with 0.20 M KOH. (note: MM of HA is 50.0 g/mol)

a. Write out the chemical formula

Answer: \_\_\_\_\_

b. Calculate the volume of KOH needed to reach the equivalence point

Answer: \_\_\_\_\_

c. Calculate the volume of KOH needed to reach half the equivalence point

Answer: \_\_\_\_\_

## Ka of Unknown Acid

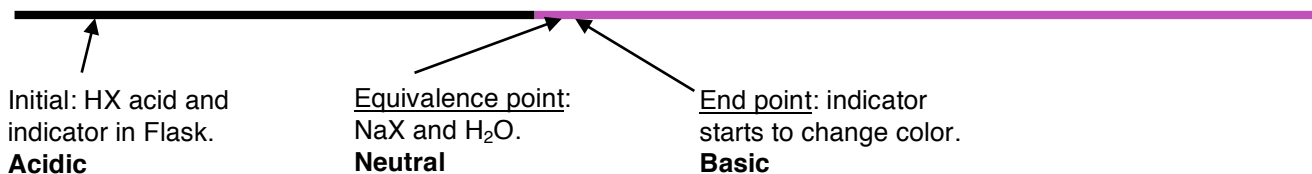
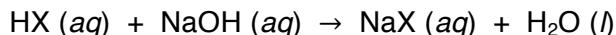
In this experiment you will determine the  $K_a$  of an unknown acid by titration with sodium hydroxide.

Because you will be titrating an unknown acid again, you will be using many of the concepts and methods learned in Titration Experiment.

When you titrate, it is important to choose the right sample size, one that requires a volume of titrant that is less than the calibrated buret volume. In our case that volume is 10 ml. Because each buret reading has an uncertainty associated with it, it is desirable to make the smallest number of readings-two- for each titration so that you minimize the error. If your sample required more than 25 ml, the buret would have to be refilled, two additional readings would have to be made for a total of four readings and additional uncertainty. In the Titration Experiment the sample size was chosen for you. In this experiment you will determine the mass of unknown acid to use by carrying out a **titration**.

### EXPERIMENT SUMMARY:

Your unknown solid is an acid. You will dissolve it in water, add some phenolphthalein indicator and then titrate to the end point with your standard NaOH solution. The **end point** is the stage in the titration at which the indicator color change is observed, indicating that the reaction is complete. The **equivalence point** is the point in titration when the number of moles of base is stoichiometrically equal to the number of moles of acid. In a titration, to get the same equivalence point as the end point, pH of the indicator should match the pH of the equivalence point. What is the difference between endpoint and equivalence point? To answer this question, in a titration, it would be better to determine when the equivalence point is reached; however, we observe the reaction completion at the end point. Take for example in the below picture representation of the titration of a monoprotic acid, which has only one acidic hydrogen to donate per molecule, HX, with sodium hydroxide (NaOH):



In the above example, at the beginning of the reaction, we only have acid (HX) in the flask. **Before** it reaches the equivalence point, we have unreacted acid and formed salt (NaX) and water. **At** the equivalence point, we only have salt (NaX) and water. At the end point, we only have salt (NaX), and base (NaOH) and water.

### Henderson - Hasselbalch equation:

The Henderson-Hasselbalch equation allows us to quickly calculate the pH or  $K_a$  of a solution based on the following weak acid equilibrium reaction:  $\text{HA} + \text{H}_2\text{O} \rightleftharpoons \text{H}_3\text{O}^+ + \text{A}^-$

Derivation:

$$K_a = \frac{[\text{H}^+][\text{A}^-]}{[\text{HA}]}$$
$$\log K_a = \log \frac{[\text{H}^+][\text{A}^-]}{[\text{HA}]}$$
$$\log K_a = \log [\text{H}^+] + \log \frac{[\text{A}^-]}{[\text{HA}]}$$

$$-\log [\text{H}^+] = -\log K_a + \log \frac{[\text{A}^-]}{[\text{HA}]}$$

**Therefore:**

$$\text{pH} = \text{p}K_a + \log \frac{[\text{A}^-]}{[\text{HA}]}$$

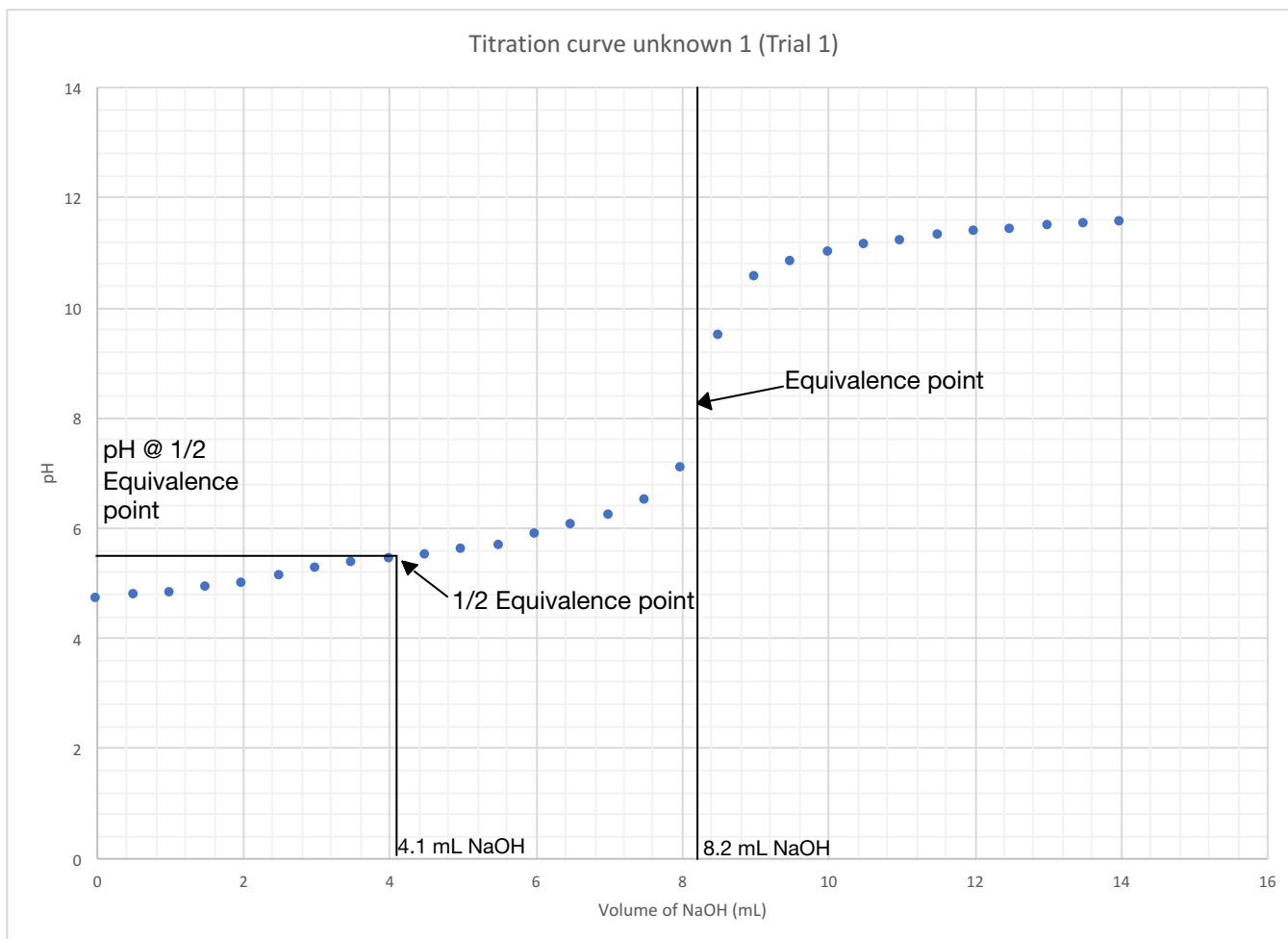
At half the equivalence point, which is when half of the amount of acid has been neutralized:  $[\text{A}^-] = [\text{HA}]$

$$\text{pH}_{(\text{half equivalence})} = \text{p}K_a + \log (1)$$

$$\text{pH}_{(\text{half equivalence})} = \text{p}K_a + 0$$

$$\text{pH}_{(\text{half equivalence})} = \text{p}K_a$$

In this experiment, since the end point and equivalence point are within the same range and are essentially the same, we can obtain the pH at half the equivalence point from a graphical plot of pH vs volume of NaOH added. As can be seen below in the graph, the end point is reached when there is a sharp increase in the pH at 8.2 mL of NaOH added. The pH at half the equivalence point can be obtained from half of the volume at the equivalence point, which is at 4.1 mL of NaOH added. By extrapolation from the data points plotted in this region onto the y axis, one can then determine the pH at half the equivalence point. From the derivation above and graphical plot, you will obtain the unknown acid equilibrium constant,  $K_a$ .



## PROCEDURE

### A. TITRATION TO DETERMINE THE SAMPLE SIZE OF UNKNOWN

1. Check out a buret and stir bar from the stockroom. Clean the buret, then rinse and fill it with your standard NaOH solution. (Don't forget to swirl the NaOH before you use it.) Take your initial reading and record it **below**; **do not** record data for this titration on your report sheet.
2. Obtain an unknown acid sample from your instructor. Weigh to the nearest 0.01 g of your unknown acid on a top-loading balance as follows:
  - a) Tare a clean dry 250 ml beaker on the top-loading balance.
  - b) Add approximately 0.25g of your unknown acid into the beaker and record below (on page 2) the initial mass.
3. Add magnetic stir bar to beaker and add approximately 25 ml of deionized water to the sample.
4. Place your beaker on the magnetic stirrer and dissolve your sample (slowly turn on the magnetic stirrer!). Add one drop of phenolphthalein to the sample just before you titrate
5. Titrate the acid solution with standardized NaOH solution, running the NaOH solution out rapidly. There is no need to be especially careful here since you need only to know the approximate volume required to titrate the sample. Don't worry about overshooting the end point a little. When you have reached the end point take your final buret reading and record **below**. If you have not reached the end point but the NaOH solution level in the buret is nearly down to the 25 ml mark, take a buret reading and then refill the buret. Take a new initial reading and then continue titrating to the endpoint. Take another final buret reading and record it **below**.

#### Mass of Unknown Sample

Sample Mass \_\_\_\_\_ g

#### Volume of NaOH in Titration

Initial reading \_\_\_\_\_ ml

Initial reading \_\_\_\_\_ ml

Final reading \_\_\_\_\_ ml

Final reading \_\_\_\_\_ ml

Volume of Titrant \_\_\_\_\_ ml



Volume of Titrant \_\_\_\_\_ ml

Using the sample mass and the volume of titrant used in the titration, calculate the mass of the sample that would require **9.5 ml of titrant**. This is the sample mass you will use in your titrations. Show a setup for your calculation below and get your **lab instructor's approval** (on your report sheet) before proceeding to part B.






Calculation:

Sample size needed to react with 9.5 mL of standard base \_\_\_\_\_

## B. VERNIER PROBE SETUP

1. Turn LabQuest 2 on and plug in the pH probe.
2. Click on **FILE SENSORS**  located on the tab top left corner.
3. Click on **MODE: TIME BASED** located on top right side.
4. Click on **TIME BASED** change it to **SELECTED EVENTS**
5. Change Name: **volume** and units: **mL** and click **OK**
6. Click on **GRAPH ICON**  located on the top right corner.
7. Your LabQuest 2 is set up

## C. TITRATION OF UNKNOWN ACID

1. Weigh one samples of your unknown acid by difference to the nearest 0.1 mg into clean 250 ml beaker.
2. Add magnetic stir bar to beaker and add approximately 100 ml of distilled water to the sample.
3. Place your beaker on the magnetic stirrer and dissolve your sample (slowly turn on the magnetic stirrer!).
4. Add one drop of phenolphthalein to the sample just before you titrate.
5. On the Labquest\_click on the  located on the bottom right corner.
6. Take the initial pH of your unknown acid solution with the pH probe by clicking KEEP BUTTON  once.
7. Place your waste beaker under your burette and fill burette 1 inch **above** the 0.00 mL marker with .10 M NaOH
8. Adjust you drop rate to 1 drop per second and allow the base level to come to the 0.00 mL marker. If you miss the 0.00 mL marker before you switch to your waste to your acids refill base and repeat step 7.
9. Switch your waste beaker with your sample beaker and lower the pH probe till immersed in solution by 1 inch (not interfering with stir bar). Titrate your unknown acid using a drop rate of 1 drop per sec.
10. Every 0.50 mL of base added to acid solution measure the pH by clicking on the **KEEP BUTTON**  until you have titrated 10 mL **past** the end point, which may be approximately 20 mL
11. Once the titration has finished hit the Stop button .
12. To Save trial
  - a. Click on filing cabinet  to change over to trial 2
13. Repeat steps 1-10 to do 2<sup>nd</sup> run

## To save your data:

- a. Click **File**, then click **save**.
  - i. Name the file.
  - ii. Click **OK**
- b. Plug your flash drive into the labquest 2
- c. Click **File**, then **export**
  - i. Click on the **thumb drive image**
  - ii. name the file
  - iii. Click on **OK**

## Importing to Excel

Open the file on your flash drive using **excel**

- i. In excel
- ii. Select **File**, then **Open**
- iii. Select **All Files** (lower right) (now you should see your file)
- iv. Select your file and click **open**
- v. Select **Delimited** and then click **Finish**
- vi. Your data should now be open in excel as a table

Using Excel Graph your data for both trials:

1. Select both rows of data and graph using **Scatter plot**.
2. Label both axis.
3. Print

REPORT SHEET: \_\_\_\_\_  
Ka OF UNKNOWN ACID

Name \_\_\_\_\_  
Last First

Instructor's initial \_\_\_\_\_

**DATA**

A. Instructor Approval of Titration to determine the sample size: \_\_\_\_\_

**B. Unknown Acid Sample**

a. **Unknown #** \_\_\_\_\_

b. **Mass of unknown acid**

i. Trial 1: \_\_\_\_\_

ii. Trial 2: \_\_\_\_\_

**C. Calculations**

a. Using your graphs accurately determine the volume of base needed to reach the equivalence point for each trial.

i. Trial 1: \_\_\_\_\_

ii. Trial 2: \_\_\_\_\_

b. Determining the pH for each trial at half the volume of base of equivalence point (half equivalence point)

i. Trial 1: \_\_\_\_\_

ii. Trial 2: \_\_\_\_\_

c. Solve for the Ka of unknown acid

$$\text{pH}_{(\text{@half equivalence})} = \text{pKa}$$

i. Trial 1: \_\_\_\_\_

ii. Trial 2: \_\_\_\_\_

d. Calculate the precision of  $K_a$

$K_a$ : \_\_\_\_\_

Theoretical  $K_a$  of unknown acid: \_\_\_\_\_ (receive from instructor)

e. Calculate the percent error:

% error: \_\_\_\_\_

#### **D. Data**

**Staple** at the end of the report, all of your labeled graphs that clearly show where you marked the equivalence point, the half equivalence point, and the pH at half the equivalence point that was used in your calculations above.