Acids, bases, and their equilibria are often characterized by examination of titration reactions. In an acid-base titration, a titrant solution of base (or acid) is added incrementally to a solution of acid (or base) in a way that allows monitoring of the extent of the neutralization reaction. Typically, this is achieved by measuring pH as a function of volume of titrant added. In lab lecture we analyze some titration curves of weak acids. No need to write a procedure for this part of lab lecture. Only write a purpose and procedure for the titration of Kombucha.

Features of Titration Curves

Changing Slope: The A^-/HA mixture (or BH^+/B mixture) present prior to the equivalence point buffers against large changes in pH even as strong acid (or strong base) titrant is added. Near the equivalence point, the buffer is destroyed as the last HA (or B) is consumed, and the pH rapidly changes. After the equivalence point, the hydrolysis of A^- (or BH^+) is suppressed by excess titrant. For the remainder of the titration, the titration curve for the weak acid (or weak base) is ~identical to that for a strong acid (or strong base).

Magnitude of the break (steep vertical region) at the equivalence point: For a weak acid, HA, titrated with a strong base, the break is smaller than for strong acid-strong base titrations. The initial pH is higher because the acid is weaker. The pH stays higher until the equivalence point because the conjugate base, A^-, formed in the titration hydrolyzes. (After the equivalence point, the hydrolysis of A^- is suppressed by the presence of excess titrant and the pH is essentially unaffected by its presence.) For extremely weak acids, the break becomes vanishingly small. Equivalence point breaks for weak bases are small for similar reasons.

Inflection point of the break: This coincides with the stoichiometric equivalence point, and its location can often be estimated visually. However, if greater accuracy is required – as, for example, when the equivalence point volume is used to calculate molar mass – the first or second derivative of the curve can be used to pinpoint it. The computer interface system used in this experiment includes software for generating the necessary derivatives.

Total Titratable Acidity (TTA) of Fermented Foods

The molecules in our food determine its taste, safety and nutrient content. Analytical chemists use quantitative analysis to analyze a food product and quantify the amount of specific molecules. For example, food samples can be analyzed via an acid-base titration to determine the amount of acetic acid in the beverage. In this lab, we will titrate a fermented food product. To simplify this lab, you will assume that the total acidity of your food product is from acetic acid and other organic acids are present in trace amounts. Total titratable acidity is a technique used in the food science and brewing industry. Acetic acid is neutralized by sodium hydroxide to produce sodium acetate and water (1).

$$HC_2H_3O_2\,(aq) + NaOH\,(aq) \rightarrow NaC_2H_3O_2\,(aq) + H_2O\,(l)$$

There is a 1:1 stoichiometry acetic acid: sodium hydroxide in this neutralization reaction. At the equivalence point the moles of base are equal to the moles of acid. Knowing the moles of acid and

volume of fermented food sample allows us to determine the total acidity of the sample as grams of acetic acid/ L of solution. Last semester we did a titration using the indicator phenolphthalein, this semester we will use a pH meter to determine the equivalence point.

Safety: *Safety goggles must be worn at all times.*

Waste Disposal: *Dispose of titration solutions (unknown plus titrant), leftover unused titrant, and leftover solid unknowns in separate waste collection containers.*

Experimental Procedure

A. Calibrating the pH Electrode - Offset Error Measurement and Measuring the pH of Kombucha

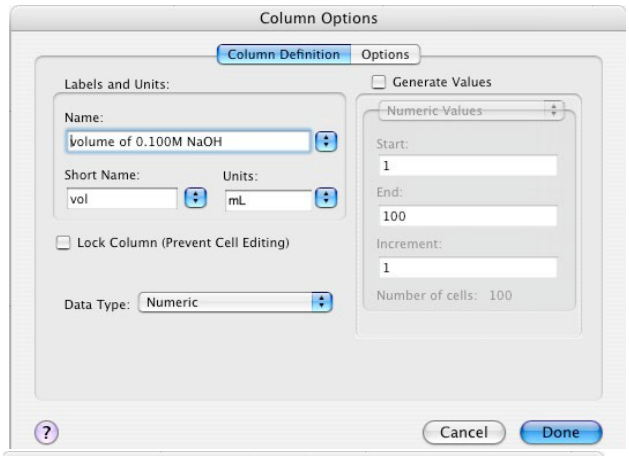
Calibrate the Vernier combination pH electrode (determine its offset error) using pH 4, 7, and 10 buffers as described in Appendix H. Record the details of the offset error measurements and calculations in your notebook. *Time saving tip: Calibration can easily be done by one person. Other group members should move on to other tasks.*

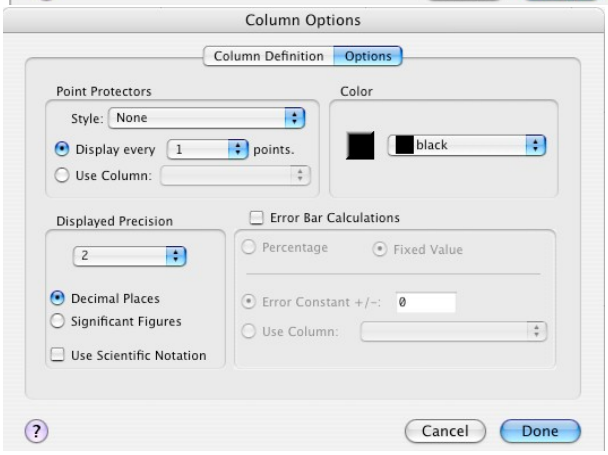
Measure ~ 20 mL of Kombucha and put in a 50 mL beaker. Measure the pH of the Kombucha and record in your lab notebook

B. **Titration of fermented food using the pH Meter/*Logger Pro***

Fill the buret with the appropriate titrant. Since the titration curve will be a plot of pH vs. *total* volume of titrant added, *be sure to adjust the liquid level to the 0.00 mL mark*. Add 10.00 mL of fermented food or beverage to a 250 mL beaker Add ~100 mL of distilled water, a few drops of indicator, and a magnetic stirring bar. Place the beaker on a magnetic stirring plate and immerse the bulb of the pH electrode in the solution. Turn on the stirrer, taking care that the stir bar does not strike the electrode.

From the toolbar, select  → **Data Collection** → **Collection** dialog box. Set **Mode** to **Events with Entry** to ensure that the pH during the titration will be recorded only upon your command (*via* a mouse click). Next, go to **Data** menu → **Column Options** → **Entry.** Through the **Column Definitions** dialog box, enter appropriate column names and units. Using the **Options** dialog box, change the displayed precision to reflect the fact that you will be estimating buret volumes to 2 places to the right of the decimal.





To begin the titration, click on 

. To record the initial pH so that it is displayed on the titration curve, click on 

and type in the volume of titrant added (0.00mL). Next, add titrant from the buret to the titration beaker, *allow time for thorough mixing so that the pH displayed in the meter window is stable*, and then record/plot the data as before.

> ***How much titrant should you add at once?*** *This varies throughout the titration. Start with ~1 mL additions since the pH is relatively flat at the beginning of the titration. In the vicinity of the equivalence point(s), however, the pH changes dramatically so titrant must be added much more slowly – even drop wise – to obtain enough points to define the shape of the pH-volume curve. The current pH is continuously updated and displayed as a dot on the graph window. Keeping track of how close the current pH is to the last plotted pH value will be useful in deciding how to adjust the rate of titrant addition.*

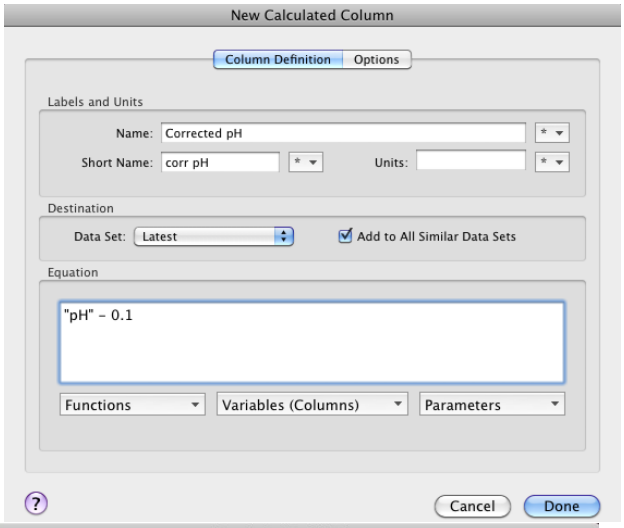
Continue adding titrant and measuring the pH and volume of titrant added to obtain the complete titration curve. Always type in the *total volume of titrant added*, not the volume of the increment most recently added. Titrate the sample until a pH a pH of ~ 9.00 is reached.

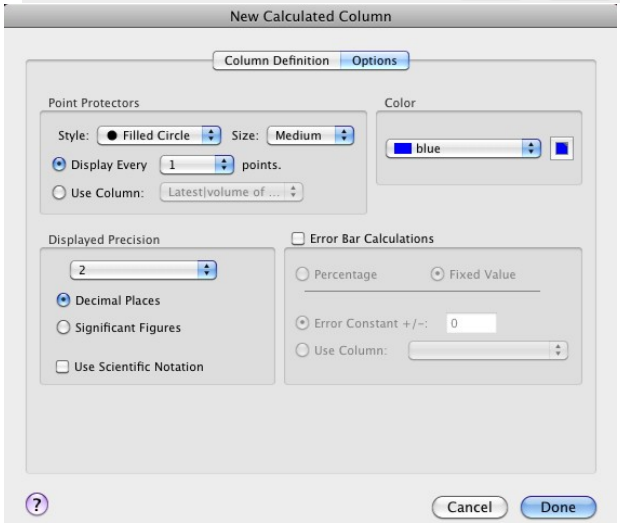
Rinse the buret thoroughly with water. Discard the *titration reaction mixture plus any leftover solid unknown* into the ***Acid-Base Waste*** collection container.

Data Analysis (DO NOT NEED TO WRITE A PROCEDURE FOR THIS PART OF THE LAB)

A. Correct the Measured pH Values for Offset Error

Go to **Data** menu → **New Calculated Column** and specify labels and an offset correction equation in the **Column Definitions** dialog box. For the example shown below, the measured pH in Part C was always about 0.10 pH unit too high, so that number is *subtracted* from the measured pH. *Note that "" marks must enclose the pH term in the equation box.* Use the **Options** dialog box to adjust the displayed precision and specify the data point style/color you prefer.





B. Locate the Equivalence Point

The total titratable acidity is defined as the volume where all the moles of acid are neutralized. For a food sample assumed to have mostly acetic acid , this volume occurs at a pH of 8.20. Using the corrected pH values determine the volume at pH 8.20 and record in your lab notebook.

Writing your Report

Calculations

- Calculate the moles of acetic acid in your sample
- Calculate the molarity of the acetic acid in your sample
- Calculate the total acidity of your Kombucha (grams of acetic acid/L)

Results

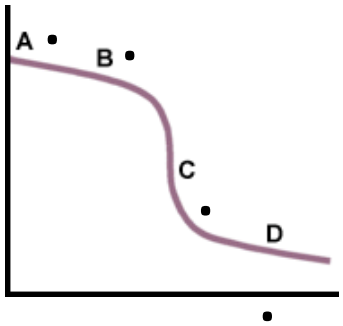
Report the pH of your sample

Report the total acidity of your sample

Questions

1. The equivalence point pH for the titration of HNO_3 with NaOH titrant is 7, but the equivalence point pH for a formic acid (HCOOH) - NaOH titration is not equal to 7. Why? Begin your answer by writing the equations for the titration reactions. Then identify the salt that must be present at the equivalence point in each titration and explain its effect on pH.

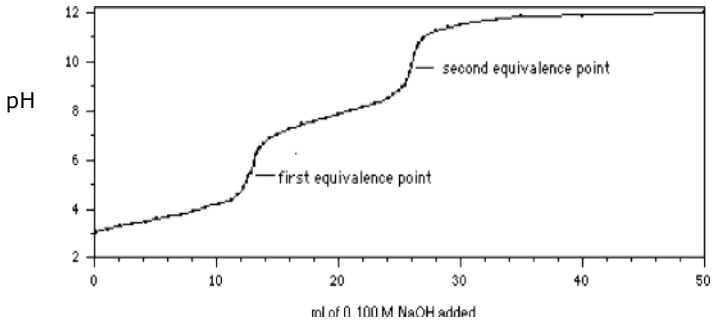
2. Here is the titration curve for a monobasic weak base, Z, with 0.100M HCl.



   a. Write the balanced equation for the titration reaction between Z and HCl.

b. What variable is plotted on the y-axis? The x-axis?
c. If the pK_b of base Z is 3.5, what is the value of pH at point B?
d. Will the equivalence point pH be <7, = 7, or > 7? Explain.
e. What is/are the major species (B, BH^+) present in solution at point D?
f. If 0.100 mole of Z is present at the beginning of the titration, what is the volume of titrant required to reach point C?

3. The diprotic weak acid whose titration curve is depicted below can be represented as H_2A.



a. Write a balanced equation to explain what happens to H_2A between the start of the titration with NaOH and the first equivalence point.
b. Write a balanced equation to explain what happens to the acid from the first to the second equivalence points.
c. Consult *Appendix K* of the lab manual for the pH intervals and color changes of indicators. Let's say that you need to find the *total moles of* H^+ present in a sample of H_2A. You don't have access to a pH meter and so must use a visual indicator to detect the endpoint of your titration. Which indicator would you choose? Why that one?