Chemistry Lab for Week 1, spring quarter.
Titration of a weak acid.

Useful reading: Section 17.3 of the text describes this experiment in good detail. This lab is designed for groups of 2-3. We will review proper use of the pH meter at the start of lab.

Introduction:

In the fall quarter, the class did a titration to determine the formula weight of an unknown acid by titration with standardized NaOH, using an indicator as a measure of the equivalence point. We will do a similar experiment in this lab, again working with an unknown acid. However, we will follow the course of the reaction by measuring the pH of the solution after each addition of base to our acid solution. We can use this information to tell us more about the acid than its formula weight. We will be able to tell if it is monoprotic or polyprotic, and we will be able to estimate the Ka of the acid.

We will provide you with a 1 M solution of some unknown acids and an already standardized solution of sodium hydroxide. Your products from the lab should be graphs showing pH as a function of amount of base added. From this graph, you should be able to determine the equivalence point for the neutralization of your unknown acid and estimate its Ka value. Most of the acids used today are fermentation products or involved in metabolism in some form or other.

Procedure:

Set up a titration setup similar to the one used in the fall. (In principle, you should just have to look at your lab notebook from fall to do this.) This will consist of a ~250 mL Erlenmeyer flask on a magnetic stir plate, with a clean burette in a burette stand positioned to allow addition of base. You will need to position a pH electrode to allow measurement of pH without the stir bar smashing into the electrode.

Both the acid solution and base solution are concentrated stocks. For both the acid and the base, make a 0.1 M working solution by dilution of the provided 1.0 M stock solutions. The stock solutions can be measure by the pump delivery systems, and the best glassware for measuring the final volume of your diluted solution is a volumetric flask.

Load your burette with your 0.1 M NaOH solution and record the starting volume. Using a pipette, transfer 25 mL of your diluted unknown acid solution to your Erlenmeyer flask and establish gentle stirring. Add the electrode from the pH meter and determine the starting pH of you acid solution.

Now begin adding NaOH solution in small increments (at the start, 1-2 mL) and record the pH after each base addition. Initially, the pH changes will be relatively small. As you proceed and when you notice a larger pH change for each addition, add smaller amounts of base. Near the equivalence point, you may find large changes of pH by adding 0.1 mL of acid. When the changes per addition are relatively small, you can then again add somewhat larger volumes of base.

When should you stop? You should make that decision based on what pH you have reached. Since it is possible your acid could be diprotic or triprotic, continue past the first equivalence point reached. Continue adding base until your solution reaches a pH of 10-11. You should repeat this procedure at least twice.

Analysis:

Prepare a graph with pH on the y-axis and mL NaOH added as the x-axis. Use the graphs in section 17.3 of the text as models. First, find any equivalence points. Where does the pH change most rapidly for a given amount of NaOH solution? You can estimate this point visually. You can also do this in a quantitative fashion by making a secondary graph in which you plot (change in pH/volume added) vs. volume and look for the maximum value for this change. Do you have evidence for more than one equivalence point? If you have multiple equivalence points, what should be true about the volume of NaOH solution added between each of the equivalence points?

At the half-way point between your starting solution and the equivalence point, the first or more acidic group will be half-neutralized. If it is half neutralized, this means that the concentrations of the conjugate acid and conjugate base forms of this acid are equal. When these concentrations are equal, the pH is equal to the pK_a of the acid in question. Use the definition of Ka to show that this relationship is true. This is also the point at which the slope (change in pH/volume added) is smallest. From your graph, estimate the pK_a of the most acid group.

If there is more than one acidic proton, the analysis method above for finding pK_a can be applied to each one. Find the half-neutralization point between each successive equivalence point, and use that to estimate the pK_a of the next acid.

In which pH ranges would your unknown acid be a good buffer?

Clean-up

When you are finished, mix any unused acid and base solutions together to neutralize them, and rinse the neutralized products down the drain. Be sure to rinse the burettes thoroughly with deionized water, and store the pH electrodes appropriately.