# Experiment 7

# POTENTIOMETRIC TITRATIONS AND MULTI-PROTIC ACIDS

**REFERENCE:** Text, Chapters 11, 12 and 15

**NOTE:** The write-up for this laboratory exercise is, like the buffer lab, different from the quantitative lab write-ups. Answer the questions posed at the end of this section in detail to hand in. **Be sure to bring a disk to lab to store data files.**

You should work in groups of two, but please submit your own, independently written, lab report.

## Potentiometric Titrations

A potentiometric titration is an experiment where, rather than using an indicator that only identifies the titration endpoint, the entire progress of the titration is monitored via a potential measurement. In this lab the pH (a converted potential) is monitored. The data that is collected is easily converted to a plot of pH vs volume of titrant — a titration curve. The equivalence point of a potentiometric titration is the point at which the curvature changes from positive to negative (the inflection point) and indicates the complete chemical conversion of the analyte by the titrant.

We will use pH electrodes, interface boxes, and laptop computers to both monitor solution pH and record and store data. Please treat the pH electrodes as the breakable and sensitive instruments they are. Always be aware that the tip of the electrode is very fragile and act accordingly. Be sure to thoroughly rinse the pH electrode with a stream of deionized water whenever you move the electrode from one solution to another, and please be careful to always leave the electrode soaking in the buffer solution you found it in. This keeps the electrode hydrated and prolongs the life of the electrode.

Although the software we use does have stored calibration values, more accurate pH readings can be achieved if we first perform a 2-point calibration of the electrode. In effect we are going to electronically store the appropriate slope and intercept values for the line that relates measured potential to solution pH. This should be done every few hours and ideally should employ buffers in the pH range of interest.

## PROCEDURE

### Setting up the equipment

1. With the computer on the bench riser and the power off, connect the LabPro interface module to the computer via the USB ports and cable. The USB port on the interface module is found on the right hand side (when looking down on the module) and is designated with a lightning rod design. The USB connection on the computer is on the right-hand-side of the computer near the back. Use the more forward of the two USB ports.

2. Connect the pH electrode to CH 1 on the left-hand-side of the interface module.

3. Connect the correct power cord to the interface module and plug into an outlet. You should hear some tones indicating the module is powered up.

4. Connect the correct power cord (the cord with the in-line surge suppressor) to the computer, plug it in and turn on the computer by lifting the monitor/lid and depressing the silver power button.

5. Log on using the computer tag number as the user name and as the password.

6. Double click the Logger-Pro icon to access the software.

7. If a dialog box comes up requesting scan information, have the software scan for the USB input. This probably will only occur the first time the software is used.

8. Go to File → Open → Chemistry with Computers → Experiment 24 → Exp 24 PH Sensor

Calibrating the pH Electrode

First Calibration Point

1. Pour pH 4.0 and pH 7.0 buffer solution into separate beakers to a depth of approximately 2 cm.

2. Choose Calibrate from the Experiment menu and then click "Perform Now".

3. Unscrew the cap on the storage buffer solution and remove the pH electrode. Remove the cap from the electrode and wash the end of the electrode thoroughly with deionized water. Do not spill or dump out the buffer solution. At the conclusion of the experiment you will replace the electrode in the solution.

4. For the first calibration point, rinse the pH electrode with distilled water, then place it into the pH 4.0 buffer.

5. Type "4" in the edit box as the pH value.

6. Swirl the electrode, wait until the voltage for Input 1 is stabilized, then click "Keep"

Second Calibration Point

7. Rinse the pH Electrode with distilled water, and place it into a buffer of pH 7.0.

8. Type "7" in the edit box as the pH value for the second calibration point.

9. Swirl the electrode and wait until the voltage for Input 1 stabilizes. Click "Keep", then click "OK". This completes the calibration.

10. You are now ready to collect data, using the calibrated electrode.

## Potentiometric Titration Number 1: Strong Base/Strong Acid Titration

### Procedure

1. Fill a buret with your standard NaOH solution (remember proper procedure!). It is convenient if you adjust your buret to 0.00 at this point. (Note: normally you would never try to adjust a buret to a set value. Instead you would read the buret at whatever level it happens to end up. However, for plotting purposes and data manipulation using this software, it is convenient to set the initial liquid level to 0.00.) Prepare the solution for titration. Pour 50 mL of deionized water into a 250 mL beaker then pipet in 25.00 mL of the HCl solution. Add a stir bar to the solution. Place the beaker on the stirrer with the buret positioned above it.

2. Clamp the pH electrode in position so that it is able to monitor the solution pH. Be sure to keep the pH electrode clear of the stir bar. If the stir bar strikes the electrode it could easily break it. **Be careful!** Begin gently stirring the solution and maintain the same stir rate for the duration of the titration.

3. To begin recording data, click on the "COLLECT" button on your computer screen.

4. Once the pH reading has stabilized, click on "KEEP". You will be asked to enter a volume reading from the buret and the software will simultaneously record the pH.

5. Add titrant at a rate anywhere between 2 and 10 drops at a time, depending on how much the pH is affected by each drop. After each addition, allow the pH to stabilize and then click on "KEEP" and enter the buret reading.

6. Continue until the titration is complete and then click on "STOP".

7. Remove the pH electrode from the solution and rinse thoroughly with deionized water and store in a buffer solution until your next titration.

8. If any data points were incorrectly recorded, now is the time to correct them on the data table.

9. Go to File and Export Data and save your data as a generic text file on your disk. This format is readable by EXCEL. Note that first and second derivative data is saved as well and should help you with equivalence point determination.

*Those of you with some calculus will be familiar with what is meant by a derivative. For the rest of you, think of the first derivative points as estimates of the slope of the original graph. The second derivative points are estimates of the slope of the first derivative graph, or "the slope of the slope" of the original graph. How might these plots help you find the equivalence point? Plot them and see if they make sense to you. Be careful — sometimes they can be misleading.*

**Potentiometric Titration Number Two: Strong Base/Weak Monoprotic Acid Titration**

**Procedure: Follow the procedure listed above except use 25.00 mL of the acetic acid solution.**

**Potentiometric Titration Number Three: Strong Base/ Weak Diprotic Acid**

**Procedure: Follow the procedure listed above except use 25.00 mL of the maleic acid solution.**

**Clean Up**

When done with your titrations, thoroughly rinse the pH electrode with deionized water and place it into the buffer-filled, sealed storage bottle. Close out of the Logger Pro software and shutdown the computer from the Start menu. Unplug the computer and LabPro module. Clean up your work area.

**FOR YOUR REPORT**

1. Print out titration curves to include in your report. On one sheet of paper please stack three plots for one titration: 1) original data (pH (y-axis) vs mL titrant (x-axis)) ; 2) 1st derivative plot; 3) 2nd derivative plot.

Label these plots as indicated below in your write-up, and refer to them as such in answering the questions.

Figure 1: Strong Acid/Strong Base titration A) Original data; B) 1st derivative C) 2nd derivative
Figure 2: Acetic Acid/NaOH titration A) Original data; B) 1st derivative C) 2nd derivative
Figure 3: Maleic Acid/NaOH titration A) Original data; B) 1st derivative C) 2nd derivative

For example, if you are answering a question for the strong acid/strong base titration using the 1st derivative plot, refer to Figure 1B. Be sure that Figure 1B is labeled properly.

**ANSWER THE FOLLOWING QUESTIONS**

1. For the strong acid/strong base titration:

   a) Find the concentration of the HCl solution using data from one of the titration curves. Show your calculation. Explain how the equivalence point volume was obtained. From which titration curve (Figure 1A, 1B, or 1C)?

   b) Explain what you expect the equivalence point pH value to be. What pH is found experimentally at the equivalence point? Mark the equivalence point on Figure 1A.

2. For the weak acid/strong base titration:

   a) Find the concentration of the acetic acid solution using data from one of the titration curves. Show your calculation. Explain how the equivalence point volume was obtained. From which titration curve (Figure 2A, 2B, or 2C)?

   b) Calculate what you expect the equivalence point pH value to be. What pH is found experimentally at the equivalence point? Mark the equivalence point on Figure 2A. (Use the text to find the acid dissociation constant for acetic acid.)

   c) Find the Ka value of acetic acid from the titration curve – Figure 2A. Show how this was done. (Hint: Use the buffer region of the titration curve).

3. For the weak diprotic acid/strong base titration:

   a) Find the concentration of the maleic acid solution using data from one of the titration curves. Show your calculation. Explain how the equivalence point volume was obtained. From which titration curve (Figure 3A, 3B, or 3C)? Check to make sure the reaction stoichiometry reflects your choice of equivalence point.

   b) Calculate what you expect the first equivalence point pH and the second equivalence point pH to be. Mark these equivalence points on Figure 3A. (Use the text to find the acid dissociation constants for maleic acid.)

   c) Find the Ka values of maleic acid from the titration curve – Figure 2A. Show how this was done.

(Hint: Use the buffer regions of the titration curve).

Note: When you use these files in EXCEL, you'll be asked to confirm the nature of the data. Just click the Next options presented. Once in EXCEL, you may want to save these files as EXCEL files, especially if you've manipulated the data in any way and/or created charts.