Potentiometric Titration of Polyprotic Acid and Base

Before doing this experiment, you should read the appropriate section in your textbook dealing with pH measurements and potentiometric titrations. You may also need to consult the sections dealing with the titration of salts, mixtures of acids, mixtures of bases, and mixtures of phosphates.

This experiment will demonstrate both the utility of the potentiometric pH titration and the characteristics of titrations of polyprotic acids. You will find that using the spreadsheet to analyze the data *in situ* will make this experiment easier and more revealing. Unfortunately the complexity of the operations and the scarcity of the equipment will necessitate working in pairs on this experiment.

Procedure

Again you will need to make some dilute (in this case 0.2 M) NaOH and HCl solutions for the titrations. As usual, 250 mL should be enough, so use your calibrated 25 mL pipet to deliver two aliquots (50 mL) of the stock solutions into your 250 mL volumetric flask. Make the NaOH first and store it in the brown plastic bottle, and then the HCl, which you can keep in the flask.

Calibrate the pH probe before starting the titrations. If you use the Vernier interface and probe, you will need to do a two point calibration with 2 pH buffers of your choice. (Refer to the section below on *Logger Pro and pH probe calibration*.) The normal pH meters in the lab are more accurate and precise, but you have to read and record the pH value manually. Most students prefer to use the Vernier probes that are interfaced to the computer so that they record the pH data automatically (you still have to read the volume from the buret).

Set up the buret and pH meter as shown in Figure 1. Depending on the pH meter you use, the glass and calomel electrodes may well be encased in one electrode, as is typical for modern designs. The glass electrode is delicate, so don't use them to stir and be careful not to hit them while you are stirring with a glass rod. The Vernier pH electrode can be used as a stirrer. For best results, the electrode(s) should be as low in the beaker as possible. Also, the buret tip should be low enough that splattering does not occur when titrant is delivered. (Note 1) Before taking each pH measurement, be sure to stir the solution in order to get a good reading.

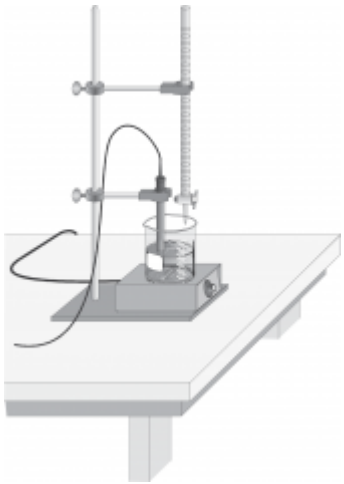
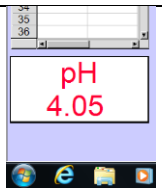
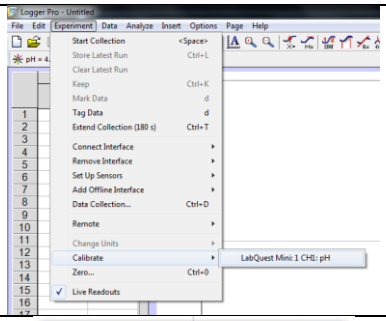
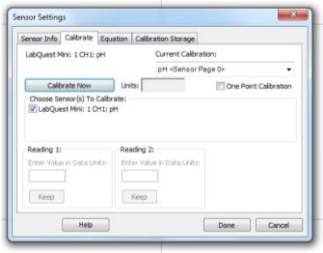


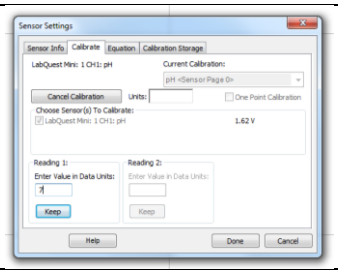
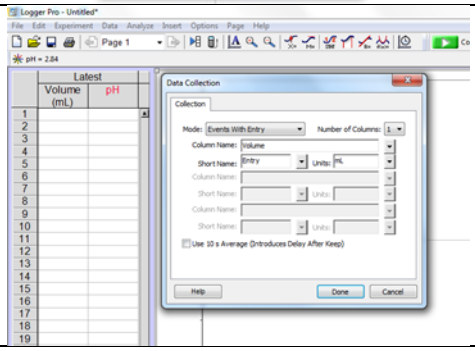
Figure 1 The pH probe set-up

Logger Pro and pH probe calibration

Open the **Logger Pro** program from the **Start** menu then plug the pH probe into the interface. The interface will recognize the sensor (if everything is right) and the box on the bottom left corner should appear with a pH reading in it

Before starting the calibration, obtain two calibration buffer solutions and a wash bottle filled with DI water for rinsing. {The pH probe is normally stored in a pH 4 buffer storage solution, but if the probe is dry when you find it, soak it in the storage solution (obtain from your TA) for 5-10 minutes before calibration to saturate the pH electrode surface.} We usually have pH 4, pH 7 and pH 10 calibration buffers available and any two buffers will be suitable for this calibration.

1. If necessary, connect the pH Sensor to your computer through the Vernier computer interface. (If you don't see the pH reading on the bottom left corner after connecting the pH probe, ask your TA for help)	
2. From the **Experiment** menu, Choose **Calibrate** and then click the **LabQuestMiniCH1: pH** flyout.	
3. A new window should appear, then click **Calibrate Now.**	
4. Remove the storage bottle from the pH Sensor; rinse the tip of the sensor with DI water, and place the sensor in the first buffer solution so the tip is immersed.	

5. Type the pH of the current buffer solution in the edit box and click **Keep**.	
6. Rinse the pH sensor with distilled water and place it in the second buffer solution. Type the pH of the second buffer solution in the second edit box and click **Keep**.	
7. Click **Done** to complete the calibration process.	
8. The titration curve and the data file can be generated from the program: under **Experiment**, click **Data Collection.**	
9. The window will open; from the drop down menu choose **Event with entry**	
10. Enter Volume (mL) as the Title, then click **Done**. (The other axis title is automatically pH.)	

Titration of Phosphoric Acid

With your volumetric pipet, measure 25.00 mL of 0.06 M (nominal) phosphoric acid into a 100 mL beaker and complete the set-up in Figure 1 to titrate with standardized 0.2 M NaOH.

Record the volume and pH every time the pH changes by 0.2 to 0.3 pH units (watch the pH meter or computer display and the buret). In the Logger Pro software, you will read the volume from the buret, enter this in the box and click the button to freeze the pH reading for that volume. In the flat part of the titration curve (where the pH is changing very slowly with added titrant) you will have to add relatively large increments of titrant, perhaps several mL, in order to get a change in pH of 0.2. However, in regions where the pH changes rapidly you will only need to add a drop. By watching the pH change rather than the volume of titrant you will collect a large number of points in the region of the equivalence point and relatively few points in the flat portions of the curve. This is a more time efficient and information-rich procedure than the indicator based titrations you have done previously. Continue to record the volume and pH until approximately 10 mL beyond the last (second) end point.

Titration of Trisodium Phosphate

Weigh out 0.24 g of solid trisodium phosphate into a 100-mL beaker. The weight must be accurate to four significant figures. Dissolve the salt in a minimal amount of DI water and titrate potentiometrically (as above) with standardized 0.2 M HCl.

When you are finished with both titrations, attach the storage bottle to the probe and screw the cap on firmly to avoid storage solution leakage.

REQUIRED MEASUREMENTS

Try to run at least two replicates of the two titrations (phosphoric acid and trisodium phosphate) so that each partner will have the opportunity to operate the buret. (The other partner should be entering the data in Excel or the Vernier software.) As usual, your accuracy and precision would be better if you could get three replicates, but this lab is time consuming due to instabilities in the pH meters, so that may not be practical. Present both (acid and base) titration curves for each operator to the TA. (Note 2) On each graph label the points (or region) that correspond to:

a) a **salt** of dihydrogen phosphate (NaH_2PO_4 (aq))
b) a **salt** of monohydrogen phosphate (Na_2HPO_4 (aq))
c) a **buffer** of dihydrogen phosphate and monohydrogen phosphate

Present first and second derivative plots for at least one of the phosphoric acid titrations to your TA. Using the derived endpoints for both phosphoric acid titrations, **calculate the concentration of the phosphoric acid solution** and report the mean value to your TA (the four values aren't from truly replicate analysis, so we won't calculate an RSD).

NOTES

(1) When setting up your apparatus it may be necessary to add a little water to the sample solution in order to cover the electrode tips. However, the end point is sharper (steeper or more sudden) for more concentrated solutions. Consequently, you want to avoid adding any more water than is necessary. Thus the 100 mL beaker is recommended for this experiment.
(2) Just as there are certain requirements for an acceptable laboratory notebook, there are also requirements for an acceptable graph. You are required to use a computer plotting program, but it is possible to produce bad graphs with a plotting program. Try to always include the following on your graphs:
a) Label both **axes** and **give the units**.
b) Add pertinent information such as the identity of the sample, sample size, concentration of titrant, etc.
c) Give a reference to the page in your notebook where the procedure can be found.
d) Your name and the date.

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