Chemistry Outreach (SVHS)
UNB Fredericton December 2014

Titration of a Triprotic Acid

Procedure

- The computers should already be set up with Figure 1 on the screen. If there is a problem with the software during the experiment please consult an Instructor.

Calibration of pH electrode (see Figure 1)

1. Add approximately 15 mL of either the pH 4 or pH 10 standard buffer solutions to a 50 mL beaker. Note the colours of these solutions so they do not become confused with each other or with the titrated solution. One bench will start with the pH 4 buffer, and the other bench with start with the pH 10 buffer, and the two benches will switch to use the other buffer. If you are starting with the pH 10 buffer make sure you enter 10.00 in box C first in step 4.
2. Remove the pH electrode from the screw-capped plastic container and place the screw-capped plastic container in a safe place away from the working area so that the contents do not spill. Push the lid of the container up the body of the electrode. If not being used for a few minutes the electrode can be placed in the Erlenmeyer flask it was originally in.
3. Rinse the lower body and the glass bulb of the electrode three times with small portions of distilled water from the wash bottle. Collect these washes in the waste beaker (800 mL). Gently dab the electrode dry with a paper towel. Immerse the electrode in the pH 4 or pH 10 buffer beaker so that the glass bulb is completely immersed in the solution. Swirl the solution for a few seconds to ensure that the bulb is completely exposed to the solution.
4. The computer software should already be opened to the calibration screen shown in Figure 1 (see above and ask for assistance if it is not). The pH that the electrode is reading is displayed in box A. Enter “4.00” in box B and click on “Read from sensor”. Make sure the value that appears in box A is approximately 4.0 and stable (not increasing or decreasing). This value will fluctuate, but not more than ±0.1 pH units. If the value in box A is not close to 4.0 and stable, then click the “Read from sensor” button until it is.
5. Remove the electrode from the pH 4 standard buffer solution, rinse it three times with distilled water and dab it dry with a paper towel. Immerse the rinsed electrode in the pH 10 standard buffer solution in the same manner as for the pH 4 standard buffer solution.
6. Repeat the sequence in step 4 of this calibration procedure for the electrode in the pH 10 standard buffer except enter “10.00” in box C (see Figure 1) and click on the adjacent “Read from sensor” button”. When this is complete click on the “Ok” in the Calibrate

Sensors window, and close the Experiment Setup window by clicking the "×" in the upper right corner (not the "×" in the upper right corner of the application window). The sizes of the Digits, Table and Graph windows can be adjusted by clicking and dragging the bottom right corner of each window, and moved by clicking a dragging the blue banner at the top of each window.

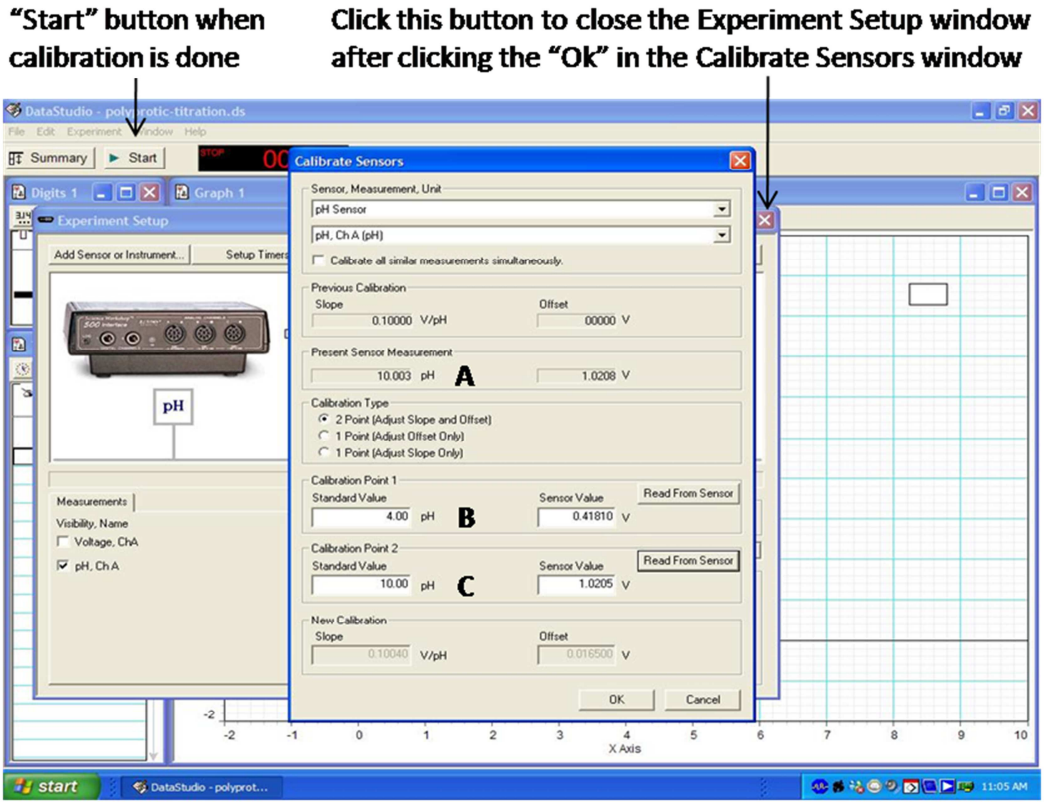

Figure 1: Calibration screen

Titration of phosphoric acid with sodium hydroxide (see Figure 2)

1. Take a 50 mL beaker to the bottle of phosphoric acid and dispense 10 mL of the solution into the beaker using the bottle top dispenser (slowly push down the top of the dispenser to dispense the solution).
2. Rinse the electrode with distilled water and dab it dry. Place the electrode into the phosphoric acid solution so that the glass bulb is completely immersed in the solution. Click on the "Start" button to begin the data acquisition. Note that the software is reading the pH of the solution (approximately pH 2). Swirl the beaker beneath the electrode, and once the pH is stable press the "Keep" in the software (see Figure 2), enter zero in the window that appears, and click "Ok".
3. The burette has already been filled with 0.100 M sodium hydroxide (titrant) to above the zero mL mark of the burette. The level of the titrant in the burette must be lowered to the zero mL mark (bottom of meniscus) before any titrant is added to the phosphoric acid

solution. Slowly rotate the stopcock at the bottom of the burette to add a drop to the waste beaker to lower the level to the zero mL mark. If you go below the zero mL mark, ask for assistance. Position the burette using the burette clamp so that is can freely dispense drops of titrant into the beaker containing the electrode and phosphoric acid.

4. By *slowly* turning the stopcock on the burette dispense up to 4 drops of titrant into the beaker of phosphoric acid. Swirl the beaker to ensure that the solution is thoroughly mixed. Observe the pH reading on the screen, and when the pH of the solution is relatively stable press the "Keep" button. Observe the level of the titrant in the burette. Enter the volume from the burette in mL (estimate the volume to the closest 0.05 mL, where each graduation is 0.10 mL) in the window that appears and click "Ok". If a mistake is made while entering the volumes ask for assistance.
5. Repeat the sequence in step 4 until a pH of approximately 11 is obtained. This process will take at least 30 minutes. The sequence of steps to follow for the titration is:
    a. Add 2 drops of titrant and swirl beaker to mix the solution.
    b. Wait until the pH of the solution is stable, and then hit the "Keep" button.
    c. Read the volume from the burette and enter the volume into the window that appears, and then hit the "Ok" button.
6. Click the button with the red square next to the "Keep" button to finish the data collection.

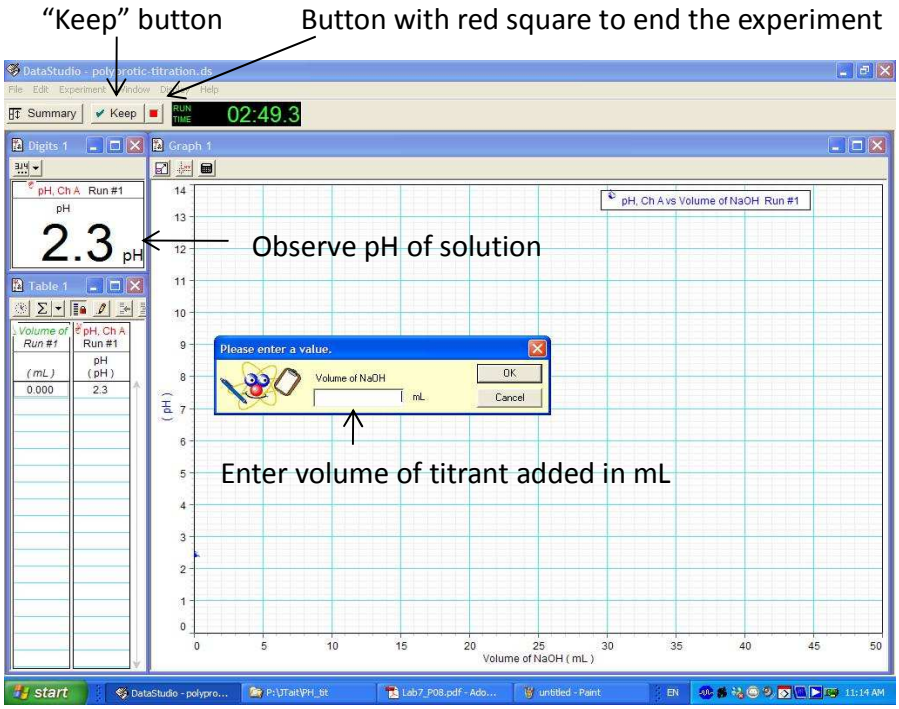

Figure 2: Collection of titration data.

Determination of the equivalence points in the titration of phosphoric acid (see Figure 3)

1. Click the button with the Calculator button at the top of the graph, followed by clicking the "Ok" button.
2. Click on the "Special" button, and then click on the "Special" button and choose "derivative(2,x)" from the menu. Click on the "Accept" button and a plot of the change in of pH (ΔpH) versus volume of titrant will appear.
3. Click on the Smart Tool button at the top of the graph (see Figure 3) and move the cursor to each of the two peaks in the ΔpH versus volume of titrant plot. Read the volume of titrant values at the tops of the peaks and record these in the data sheet at the end of this document.
4. Use a calculator (ask for assistance if you do not have one) to determine how many moles of sodium hydroxide were added at each equivalence point based on the corresponding volume of titrant added. Remember that the first equivalence point (lower volume of titrant) corresponds to *one* equivalent of titrant added to the acid, and that the second equivalence point (higher volume of titrant) corresponds to *two* equivalents of the titrant added to the acid.
5. Use a volume of 10 mL (0.010 L) for the phosphoric acid solution to calculate the concentration in molarity units (mole L^{-1}) of the initial phosphoric acid solution based on each equivalence point. Record these values in the data sheet at the end of this document.

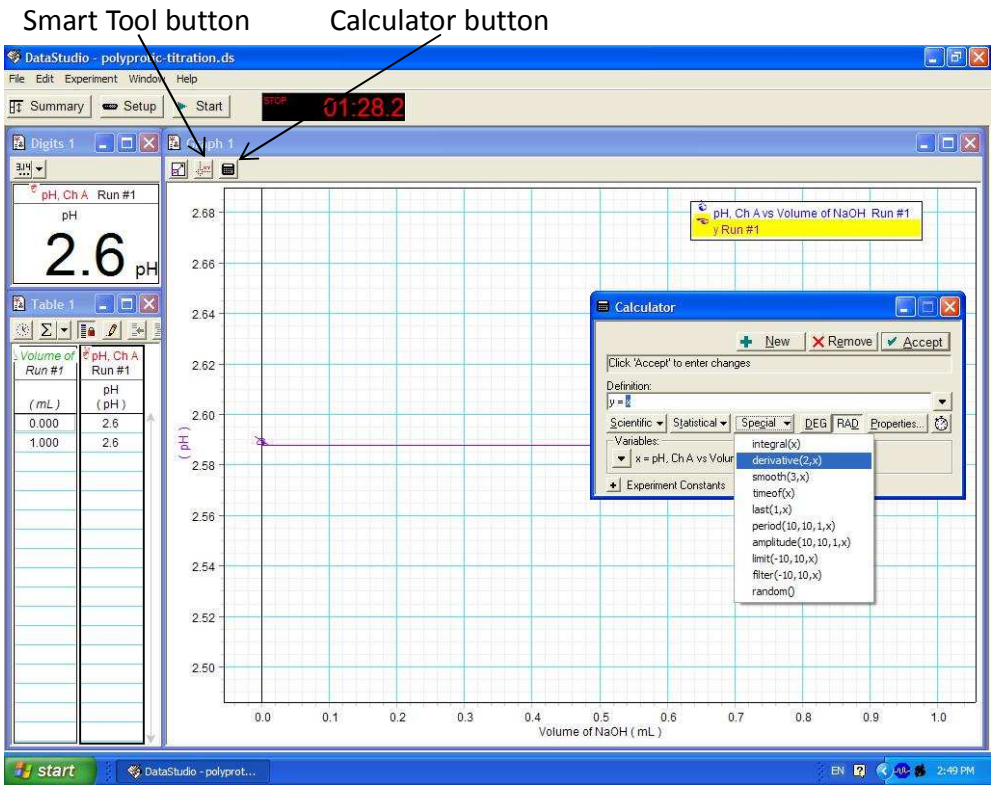

Figure 3: Calculation window at end of titration.

Determination of the pKa for the second ionization of phosphoric acid

1. Use the previously determined volumes of titrant at which the first two equivalence points occurred to calculate the *second* half equivalence point of the titration curve. The second half equivalence point corresponds to the average of the two volumes of each of the equivalence points (see the data sheet).
2. Use the "Smart Tool" to determine the pH on the titration curve that corresponds to the second half equivalence point, and enter this value into the data sheet. This value is the experimentally determined pK_a for the second ionization of phosphoric acid.

Clean up

1. The pH electrode can be removed from the titration solution, rinsed with distilled water and dabbed dry with a paper towel. The electrode should be placed back in the screw capped plastic container from which it was originally obtained. Make sure the screw is tight so the solution in the vial does not leak out.
2. The titration, standard buffer and waste solutions can all be poured in the container labelled "Aqueous Waste". The glassware should be rinsed with distilled water (these washes can be poured down the drain) and left to dry upside down on paper towels.
3. The burette should be rinsed with approximately 10 mL of distilled water three times. The burette can be leaned over a sink to ensure that these washes coat all of the inner surface of the burette. The washes can be drained through the open stopcock to rinse the tip of the burette. The burette should be clamped upside down on a paper towel with the stopcock open.

Data Sheet

Names: _____

	Values	Units
Determination of Equivalence Points and Concentration of Phosphoric Acid Solution		
Volume of Titrant at First Equivalence Point		mL
		L
Moles of 0.100 M Sodium Hydroxide Added at First Eq. Point		mole
Concentration of Phosphoric Acid Solution		M (mole L^{-1})
Volume of Titrant at Second Equivalence Point		mL
		L
Moles of 0.100 M Sodium Hydroxide Added at Second Eq. Point		mole
Concentration of Phosphoric Acid Solution		M (mole L^{-1})
Average Concentration of Phosphoric Acid Solution		M (mole L^{-1})
Determination of the pK_a for the Second Ionization of Phosphoric Acid		
Volume of Titrant at Second Half Equivalence Point		mL
pH at Second Half Eq. Point		
pK_a for Second Ionization of Phosphoric Acid		