Lab 5: Phosphoric Acid in Cola

The "main goals" of this lab are:

1) To determine the concentration of H_3PO_4 in Cola

2) To determine K_{a1} and K_{a2} for H_3PO_4 .

In addition to the main goals there are a few important concepts that can be observed in this lab.

- 1) The shape of a polyprotic acid titration curve, revealing stepwise removal of protons.
- 2) The % dissociation of H_3PO_4 initial vs. the % dissociation of $H_2PO_4^{1-}$ at the 1st equivalence point.
- 3) The first equivalence point is when all H_3PO_4 present (both dissociated and nondissociated) has been converted to $H_2PO_4^{1-}$.
- 4) There is more than one way to use titration data to calculate K_a .
- 5) A second equivalence point for polyprotic acids can be used to calculate M of an acid, and compared to M values obtained with the first equivalence point
- 6) A third equivalence point for H_3PO_4 cannot be seen in a titration curve.

You will quantitatively titrate 0.010 M NaOH into a known volume of cola. The change in pH vs. volume of NaOH added will be monitored the using a digital pH meter. Your data will allow you to calculate the molarity of Phosphoric Acid (H_3PO_4) in the sample of cola.

Data tables and titration curves you collect will show two distinct endpoints: One for the neutralization of a single proton from H_3PO_4

(1)
$$H_3PO_4(aq) + OH^-(aq) \rightarrow H_2PO_4^-(aq) + H_2O(l)$$
 K_{a1}

And a second for the neutralization of single proton from H₂PO₄¹⁻

(2) $H_2PO_4^{1-}(aq) + OH^{-}(aq) \rightarrow HPO_4^{2-}(aq) + H_2O(l)$ K_{a2}

For reaction (1): 1 mole OH⁻ will neutralize 1 mole H_3PO_4 For reaction (2): 1 mole OH⁻ = 1 mole $H_2PO_4^{-1-}$ (equal to 1 mol H_3PO_4 initial) TOTAL mole of OH⁻ to reach 2nd equivalence point (from beginning) = 2 x mol H_3PO_4

The moles OH^- required to reach each of these equivalence points can be calculated from the Volume and Molarity of OH^- solution. With a known initial volume of cola sample, the concentration of Phosphoric acid can then be determined. Once you have established the molarity of H_3PO_4 in Cola, you will be ready to calculate mg of Phosphorous in a 6 ounce serving of Cola (main goal 1).

Your initial pH (before any addition of OH⁻) and the molarity of H_3PO_4 will allow you to calculate K_{a1} using the same process as would in solving a typical weak acid:

(3) $K_{a1} = x^2/(M-x)$ where $[H^+] = x$ and is derived as 10^{-pH} (Part of main goal 2)

After the first equivalence point has been achieved, you will continue titrating to reach a second equivalence point. An experimental determination of K_{a2} can be made by rearranging the equilibrium expression to isolate $[H^+]$:

(4)
$$[H^+] = \text{Ka}\left(\frac{[HA]}{[A^-]}\right)$$

Notice that when $[HA] = [A^{-}]; K_a = [H^{+}].$

In the K_{a2} equation shown previously, (2), $HA = H_2PO_4^{1-}$ and $A^- = HPO_4^{2-}$

Thus: $K_{a2} = [H^+]$ when $[H_2PO_4^{-1}] = [HPO_4^{-2-}]$

This is the point when half the "acid" (H_2PO_4) has been converted to "base" (HPO_4^2) . This is also known as the "half equivalence point."

Identify on your graph the volume of OH^- added which is $\frac{1}{2}$ way between equivalence point 1 and equivalence point 2. The observed pH at that volume = pK_{a2} ! (Now you've finished main goal 2!).

You will continue titrating after you see the 2^{nd} end point until you see the final pH begin to level off. But you will find that the 3^{rd} endpoint for H_3PO_4

$$(5) \mathrm{HPO_4}^{2^-} \rightleftarrows \mathrm{PO_4}^{3^-} + \mathrm{H^+}$$

cannot be detected using a pH meter. The reason for this can be partially explained by referring you to Figure 15.9 on page 606 in your text. K_{a3} for H_3PO_4 is 4.8 x 10^{-13} . From the chart in your text you can see that the endpoint is not observed for compounds with initial concentrations of 0.10M when the $K_a < 10^{-10}$.

During class time we will calculate of the true equilibrium constant for the third proton neutralization (reaction (6) below) to provide a more quantitative reason for the absence of the third equivalence point in your data.

(6) $\text{HPO}_4^{2-}(aq) + \text{OH}^-(aq) \leftrightarrows \text{H}_2\text{O}(l) + \text{PO}_4^{3-}(aq)$

You should now be able to identify whether a titration curve is for a monoprotic or a diprotic acid, and whether the acid is a strong acid or a weak acid all based on the shape of the curve.

You will calculate the % dissociation of the acid initially and at the 1st endpoint. You may wish to use this data as you respond to a post-lab question that has you suggest why the method of pH at $V_{1/2} = pK_{a1}$ does not agree with the literature value as well as the same method when used to determine K_{a2} . Consider what conditions would be necessary for this this method to be used to determine K_a for most weak acids.

Procedure:

Set up Logger-Pro to collect data using experiment titled "35 Phosphoric Acid." Connect the pH meter to the interface and calibrate your pH meter using the pH 4 and pH 7 calibration buffers. After the meter is calibrated secure the probe so it is immersed in distilled water while you prepare your solution for titration (do not allow your pH probe to "dry out.")

Use a clamp to attach a buret to a ring stand. Rinse and fill the buret with 0.010 M NaOH to the level above 0.00mL. Draw some NaOH solution through the buret to bring the level to precisely 0.00 mL, which will also ensure that the buret tip is filled. <u>Record the</u> <u>concentration of NaOH</u> in your table.

Cola contains H_2CO_3 in addition to H_3PO_4 which could affect pH readings, giving erroneous results for this lab. You are going to heat the cola in order to drive the following reaction forward:

 $H_2CO_3(aq) \rightarrow CO_2(g) + H_2O(l)$

Put approximately 100 ml of Cola into an Erlenmeyer flask. Mark the initial liquid level, and cover the flask with a watch glass. Heat the Cola at just BELOW a boil, for approximately 20 minutes to drive off the CO_2 from the Cola. After 20 minutes, allow the Cola to cool, and then add distilled water if necessary to reestablish the liquid volume to its original, marked, level.

Rinse a 10 mL pipet with your cola sample and finally draw two 10mL samples of cola (20.00 mL = total sample size) and place into a clean dry 100 mL beaker. <u>Measure and</u> record the initial pH of the Cola. Place the beaker on a magnetic stir plate, add a magnetic stir bar and additional 10mL water to the cola if necessary to insure there is enough solution for the stir bar to be immersed. <u>Measure and record the temperature of the diluted cola</u> solution.

Use a clamp to secure the pH meter with the probe in the cola solution. Don't allow the magnetic stir bar to hit the pH probe, as it will break it!



Start the magnetic stirrer and check the pH. You are now ready to perform the titration. This process goes faster if one person manipulates and reads the buret while another person operates the handheld and enters volumes.

a. Start data collection.

- b. Before you have added any NaOH solution, click Skeep and enter 0 as the buret volume in mL. Click ok to store the first data pair for this experiment.
- c. Add 0.5 mL of NaOH solution. When the pH stabilizes, click **G** Keep and enter the current buret reading in mL. Click **OK**. You have now saved the second data pair for the experiment.
- d. Continue to add 0.5 mL increments, entering the buret level after each increment. When the pH has leveled off (near pH 10), click **stop** to end data collection.

Examine the data on the graph of pH *vs.* volume to find the *equivalence point*—that is, the 0.5 mL volume increment that resulted in the largest increase in pH. As you move the pointer, the pH and volume values of each data point are displayed below the graph. Go to the region of the graph with the large increase in pH. Find the NaOH volume (in mL) just *before* this jump. Record this value in the data table. Then record the NaOH volume *after* the 0.5 mL addition producing the largest pH increase.

Print a copy of the graph of pH vs. volume. Then print a copy of the NaOH volume data and the pH data for the titration.

Dispose of the beaker contents as directed by your instructor. Rinse the pH sensor and return it to the storage solution, upright.

Cola titration data table

Temperature of Cola at beginning of titration	°(C
Concentration of NaOH	M	
	Trial 1	Trial 2
Exact volume of Cola (before diluting with water)		
pH of Cola BEFORE ADDING H ₂ O (for K _{a1} calculation)		
Volume of NaOH required to reach 1 st equivalence point		
Volume of NaOH required to reach 2 nd equivalence point		
Molarity (experimental average) of H ₃ PO ₄ in Cola		
Molarity of Cola calculations:		

Calculations for values listed on next page

Calculation for K_{a1} based on initial pH and initial [H₃PO₄]:

Calculation of K_{a1} using pH at the half-equivalence point:

Calculation of % dissociation of H₃PO₄ (in initial sample before titration):

Calculation for % dissociation of $H_2PO_4^-$ (at the 1st equivalence point):

Calculation of K_{a2}:

	Trial 1	Trial 2
Molarity of H ₃ PO ₄ in Cola		
Initial pH reading (BEFORE ADDING H ₂ O)		
% dissociation of H_3PO_4 (In sample before titration)		
Calculated value of K _{a1} From initial pH and initial [H ₃ PO ₄]		
Average calculated K _{a1}		
Experimental value of K_{a1} based on $\frac{1}{2}$ -eq. point		
Literature value of K _{a1}		
Molarity of H ₂ PO ₄ ⁻ at 1 st equivalence point		
pH at 1 st equivalence point		
% dissociation of $H_2PO_4^-$ at 1^{st} equivalence point		
Volume at 1 st equivalence point		
Volume at 2 nd equivalence point		
Volume at $pH_{1/2}$ (between 1^{st} and 2^{nd} equivalence pts)		
$pH_{1/2}$ (between 1^{st} and 2^{nd} equivalent points)		
K _{a2}		
Average K _{a2}		
Literature value of K _{a2}		

Lab 5 Post-Lab Questions

1.) A carbonated beverage maker claims that none of their colas contain more than 30 mg of phosphorus per 6-oz serving. Calculate the mg of phosphorous in a 6 - oz serving of cola, according to your experimental data.

2.) Consider the % dissociation for H_3PO_4 in the initial sample of cola, and the % dissociation of $H_2PO_4^{1-}$ at the 1st equivalence point. Use these two calculated values to explain why K_{a1} calculated using the $V_{1/2}$ method may not be valid, but K_{a2} calculated using this method is valid. Is this consistent with your experimental values for K_{a1} and K_{a2} , when compared with the literature accepted values? What general conditions must be true for the $V_{1/2}$ method to be a valid way of calculating any weak acid K_a ?

3.) There are several possible sources of error in this lab. Discuss possible sources of error you felt may have been significant in your results, as well as any specific problems or successes you had with this lab.

Lab 5 Pre-lab questions:

1.) Use the axes below to roughly sketch the titration curve you would expect for a titration of a strong acid with a weak base, a weak acid with a strong base, and for a diprotic acid with a strong base:



2.) A 20.00 mL sample of a diprotic acid H_2A was titrated with 0.100 M NaOH. The first equivalence point was reached after the addition of 14.98 mL of base. The second equivalence point was reached after the addition of 30.03 mL. Use these two data points to calculate two values for the molarity of H_2A in the original sample. What is the average experimentally determined Molarity?

3.) The pH after addition of 7.49 mL of 0.100 M NaOH was 3.70, and the pH after the addition of 22.52 mL NaOH was 7.10. Use these data points to calculate K_{a1} and K_{a2} for H_2A .

4.) The initial pH of the sample (before NaOH addition) was 1.91. Use this value to calculate K_{a1} . Does this agree with K_{a1} calculated above using $V_{1/2}$?