**Experiment 10: TITRATION OF A COLA PRODUCT**

**Purpose:** The mass percent of phosphoric acid in a Cola product is to be determined.

**Introduction:** You might have heard of the claim that “Coca-Cola takes the rust off of nails.” There is probably truth to it because of the acid content in the Cola. The acid provides a taste that is both sweet and sour, and does not interfere with other tastes in the drink. Phosphoric acid is the acid that is present in all Colas, but the percentage of phosphoric acid may vary (they don’t give out the Coke or Pepsi formula to just anyone!). A similar experiment could be performed titrating “unColas” such as 7-Up, Sprite or Squirt. In those sodas the acidity comes from citric acid. In this experiment you will titrate a sample of a Cola product and use the information to determine the percent phosphoric acid in the sample.

Colas are also carbonated beverages. The carbonation can produce some carbonic acid in the Cola, which would affect your results. Therefore, to ensure that you are only titrating the phosphoric acid you will use de-carbonated soda. This can easily be obtained by gently heating the soda, or as you may already know, by rapidly shaking the bottle. It is also important that we do not use diet Colas since the artificial sweeteners that they contain have acidic functional groups that will also interfere with the titration.

You should remember from previous titrations that the titration is complete when you reach the equivalence point. The equivalence point is when starting material has completely reacted. Usually this is not visually apparent without special aid. In past experiments you probably used an indicator that changed color very close to the equivalence point to signal when the reaction is complete. This visual change is the endpoint. In this experiment, the Cola is dark brown and would mask any color changes thus preventing the use of such indicators.

Phosphoric acid is a weak, triprotic acid. It dissociates as shown in Equations 1-3.

\[
\begin{align*}
H_3PO_4(aq) & \rightleftharpoons H_2PO_4^- (aq) + H_3O^+ (aq) & \text{Equation 1} \\
H_2PO_4^- (aq) + H_2O (\ell) & \rightleftharpoons HPO_4^{2-} (aq) + H_3O^+ (aq) & \text{Equation 2} \\
HPO_4^{2-} (aq) + H_2O (\ell) & \rightleftharpoons PO_4^{3-} (aq) + H_3O^+ (aq) & \text{Equation 3}
\end{align*}
\]

Since each dissociation of an $H^+$ occurs in a separate reaction, each reaction has its own equilibrium constant, and therefore its own equivalence point. Figure 10.1 shows what a typical titration curve for a Cola sample looks like. You should notice that although there are three equivalence points, only two can be seen on the graph. Since glass electrodes measure $H_3O^+$ ions and above pH 10.5 the acid content is very low, the electrode begins to become bombarded with the high concentration of other ions that have been added during the titration (mainly $Na^+$ in this case). This is referred to as the alkaline error, and makes the pH appear lower than it really is. For this reason we will stop the titration at a pH of 10. In so doing we will not be able to observe the third equivalence point of phosphoric acid.
The equilibrium constant for each reaction is listed below.

<table>
<thead>
<tr>
<th></th>
<th>Phosphoric acid</th>
<th>Citric acid</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K_1$</td>
<td>$7.11 \times 10^{-3}$</td>
<td>$7.44 \times 10^{-4}$</td>
</tr>
<tr>
<td>$K_2$</td>
<td>$6.32 \times 10^{-8}$</td>
<td>$1.73 \times 10^{-5}$</td>
</tr>
<tr>
<td>$K_3$</td>
<td>$7.10 \times 10^{-13}$</td>
<td>$4.02 \times 10^{-7}$</td>
</tr>
</tbody>
</table>

There are four points highlighted in Figure 10.1. Points 1 and 3 are midway in the region where the pH is relatively unchanging. From your understanding of buffers, the pH’s at these points are the pKa’s of the first and second ionization of the phosphoric acid (Equations 1 and 2). Points 2 and 4 are located in the regions of rapid pH change. Rapid pH change occurs when that acid species has just been completely titrated. Therefore these points are the equivalence points.

Figure 10.2 illustrates how to extrapolate data from your titration curve. The volume of base used to reach the equivalence point is read off the graph. Together with the given molarity of the base we can determine how many moles were used to titrate the Cola. From the number of moles of base, the number of moles of phosphoric acid can be obtained stoichiometrically. Remember that at the first equivalence point, there is a 1:1 mole ratio of NaOH to phosphoric acid, but at the second equivalence point, you have completely titrated the $H_3PO_4$ and the $H_2PO_4^-$. So at equivalence point 2 there is a 2:1 mole ratio of NaOH to phosphoric acid. The molarity of phosphoric acid is then calculated by dividing the number of moles of phosphoric acid by the volume of the Cola used in the titration.

**Safety Precautions:** Keep your safety goggles on at all times. The Cola drink is harmless, but the NaOH is not. It causes irritation to the eye and skin.

**Procedure:** Work with one partner.

1. Use a 25.00-mL pipet to deliver exactly 25.00 mL of room temperature, de-carbonated Cola into a 250-mL beaker. Check out a magnetic stir bar and gently add it to the beaker. Place the beaker on a stir plate. Set up a pH meter so that the probe is
completely immersed, but in a way that the stir bar will not hit it. *The tip of the probe has a thin glass membrane. Handle it with care!* Position the beaker so that the stir bar is as close to the center of the beaker as possible for optimal stirring. It should stir rapidly enough to ensure thorough mixing but not so rapidly as to cause splattering.

2. Obtain about 75 mL of 0.0100 M NaOH in a beaker. Use this NaOH to fill a buret. Remember that you should always rinse a buret 2-3 times with the solution that you are about to put into it and remove air bubbles at the buret tip. If you do not remember how to use a buret, ask your instructor. The initial level of the NaOH should be at exactly 0.00 mL. Once the buret is ready, place it over the beaker containing the Cola.

3. Follow the procedure in Appendix 5 on the use of the pH meter. Calibrate with pH 4 and 7 buffers.

4. Set up an Excel spreadsheet on the laptop to record the data. Your laptop should be equipped with a flash drive to record your data. If your laptop does not have one, inform your instructor.

5. In Cell A1 type “Vol NaOH” and Cell B1, “pH”. Next, highlight Columns A and B from Cell 1 through 40, and then select Insert, Scatter, Scatter with Smooth Lines and Markers. Remove the Legend from the graph. You are now set up to track the progress of your titration on the graph. Each data point you type into the spreadsheet will immediately show up on the graph. After you have completed the titration you can go back and fix up the scale maximum and minimum, titles, etc.

6. One partner will be adding the NaOH and calling out the buret reading. The other partner will read the pH and record both data directly on the laptop. (You will not be writing anything into your lab notebook.) SET UP THE FILE TO SAVE on the flash drive provided, in the folder titled “Chem 124.” Save your file as “Ch 124 Titr Cola LAST NAMES OF YOU AND YOUR PARTNER”. Use your last names. Do not use initials. Get help from your instructor or lab assistant if necessary. You will want to save periodically throughout the titration so that you don’t lose any data in case the system crashes.

7. You are now ready to titrate the Cola. Your goal is to slowly open the stopcock and allow about 0.5 mL to fall into the flask. After each addition, record the exact buret reading and the pH reading. In reality, slightly more or less than 0.5 mL can be added each time, but you must record the exact buret reading each time. This titration is more time consuming than using an indicator, but you only need to do it once (unless you make major errors). Unlike previous titrations you may have performed, you should not be rinsing the buret tip or the side of the flask with deionized water during the titration. Any added water would directly change the pH that you are measuring.

8. When you have added 40 mL of NaOH or when the pH reaches 10, you can stop collecting data. Remember to save your data before continuing. Clean the magnetic stir bar with water and RETURN IT TO YOUR INSTRUCTOR.

9. Determine the density of the Cola. You had developed a procedure for determining the density of a bleach sample in Experiment 5. Refer to your notes in your lab notebook as to how it was done. Do not repeat any mistakes you might have made in the previous experiment. Record the necessary data in your notebook. Hint: Why is it inappropriate to measure the density of 1 mL of Cola?
FINAL PREPARATION OF THE TITRATION CURVE:
You and your partner will work together to fix up the titration curve and then print one copy for each of you. Both of you are held responsible for the appearance of the graph, and a significant portion of the grades is based on it. Follow instructions below carefully.

This graph is different from other ones you have created this semester as there is no trendline involved. Instead you will focus on selecting an appropriate scale for the x-axes. Have your instructor check your graph and spreadsheet before you print them.

1. If you have not done so yet, remove the Legend. The Legend is pointless when there is only one series and the Legend takes up space unnecessarily. This is done by clicking on the Legend and pressing Delete.

2. LABEL GRAPH: Highlight the graph (by clicking anywhere on the graph), select Chart Tools, Layout, Chart Title, Above Chart and type in a title for the graph followed by names of you and your partner:
   pH versus Volume NaOH added (in mL) (Names of you and your partner)

3. LABEL AXES: Select Chart Tools, Layout, Axis Title, Primary Horizontal Axis, Title Below Axis and label the x-axis. Go to Primary Vertical Axis, Horizontal Title and type in a title for the y-axis:
   X-axis is Volume of NaOH added (in mL). Y-axis is pH.

4. ADJUST SCALES FOR X-AXIS: The x-axis needs to have finer graduation so that the volume at the equivalence points can be read more precisely. Go to Chart Tools, Layout Tab, Axes, Primary Horizontal Axis, More Primary Horizontal Axis Options (at the very bottom). In this new window, under Major, Fixed, enter 1. Under Minor, Fixed, enter 0.5. Under Minor tick mark type, select Outside.
   You will notice some of the numbers are illegible because they run into each other. We need to rotate them so we can read them. In the same Format Axis window you are at, select Alignment (on the left), Text direction, and select Rotate All Text 270°. Close.

5. GRIDLINES: We need some gridlines to help us read the volume, so go to Chart Tools, Layout Tab, Gridlines, Primary Vertical Gridlines, and select Major Gridlines.

6. We don’t need to do anything with the y-scale because we are not reading the pH off the chart, just the volume off the x-scale.

7. ADJUST THICKNESS OF CURVE AND MARKERS: We want to make the curve thinner and the points smaller so that we can get more precision out of the graph. Go to Chart Tools, Layout and on the top far-left, there is a window that by default says “Chart Area”. Use the drop down window to select “Series 1” and then below that, click on Format Selection. In the new “Format Data Series” window, select Marker Options, select Built-in and change size to 2. Next, in the same “Format Data Series” window, select Line Style. Under Width, select 0.25 pt. Close. The titration curve should now be of a reasonable thickness.

8. HIGHLIGHT THE GRAPH BY CLICKING SOMEWHERE ON THE GRAPH. Select Page Layout, Orientation, Landscape. Double check that you still have the graph
highlighted. If you are not sure, click AGAIN on the graph, then on the Microsoft button and Print.

9. PRINTING THE SPREADSHEET: Since you did not record the data in your lab notebook for this experiment, the spreadsheet is the only record you have of your data. First, make sure the spreadsheet is on Portrait Mode. Click once on Cell A1, then on Page Layout, Orientation, and select Portrait. Next, highlight Cells A1 and B1 all the way down to the last cells that contain data. DO NOT HIGHLIGHT THE ENTIRE COLUMNS, ONLY THE CELLS THAT CONTAIN DATA. Click Print Area, Set Print Area. This allows you to print just the data and not the graph. Click on the Microsoft Button, and Print two copies, one each for you and your partner.

10. SAVE AGAIN! You are now going to save both the spreadsheet and your graph into the folder designated by your instructor. You may wish to send a copy of this by email to yourself, or save it onto your flash drive if you have one with you.

OBTAINING INFORMATION FROM THE GRAPH: To be completed INDIVIDUALLY and before you leave the lab. You will need a sharp pencil. An electric pencil sharpener is provided in the lab. Refer to Figure 10.2 as a guideline.

11. Draw the two baselines in the region where the pH is increasing only gradually. Then draw a tangent to the points in the region where the pH is rising rapidly (shown as dotted line in the Figure 10.3). Find the intersections of the two baselines and the tangent at the first equivalence point. Measure the distance with your ruler, then calculate the midpoint of this tangent (between the intersections). Read the volume as precisely as you can on the x-axis (to the closest 0.1 mL). This is the volume of NaOH needed to reach the first equivalence point. Record this on your graph.

12. Repeat for the second equivalence point. The volume is the volume of NaOH needed to reach the second equivalence point (to neutralize TWO moles of H⁺ per mole of H₃PO₄).
EXPERIMENT 10: TITRATION OF A COLA PRODUCT

UNDERSTANDING EQUIVALENCE POINTS FROM THE TITRATION CURVE

In your General Chemistry I Lab you should have performed titrations of an acid (such as HCl or acetic acid) with a strong base (such as NaOH), and with an indicator (such as phenolphthalein. At the point when the solution turned from colorless to light pink, you would have added just enough NaOH to neutralize all of the acid. The change in color signifies your reaching the equivalence point.

In this experiment, by drawing the baselines and tangents, you have determined the equivalence point without the aid of an indicator. For the first equivalence point, you have added just enough NaOH to react with all of the H$_3$PO$_4$ to form H$_2$PO$_4^-$ . For the second equivalence points, there is enough NaOH to react with all of the H$_3$PO$_4$ to form HPO$_4^{2-}$.

CALCULATIONS: Show your calculation setups carefully. Do individually.

1. Write the net ionic equation for the reaction between H$_3$PO$_4$ and NaOH leading to the first equivalence point.
2. Using the volume of NaOH at the first equivalence point calculate the molarity of the phosphoric acid in the cola sample.
3. Repeat using the volume of NaOH at the second equivalence point.
4. Calculate the average molarity of the phosphoric acid.
5. Calculate the density of the Cola sample. You will need this for the next step in the calculation.
6. Using the average molarity of the phosphoric acid, calculate the average mass percent of phosphoric acid in the Cola sample.

Pre-Lab Assignment:

1. There is no need to prepare a data table for the titration ahead of time in your lab notebook as you will be entering the data directly on the Excel spreadsheet on a laptop. However, you should prepare your lab notebook for recording other data. You should know what they are by reading the procedure carefully.

2. Describe precisely in a short paragraph how you will determine the density of your Cola sample. Specify which and what size equipment you will use.

3. The phosphoric acid in a 100.00-mL sample of Cola drink was titrated with 0.1025 M NaOH. If the first equivalence point occurred after 16.11 mL of base was added, and the second equivalence point occurred after 32.55 mL of base was added, calculate the molar concentrations of H$_3$PO$_4$ in the Cola sample based on (a) the first equivalence point, and (b) the second equivalence point.

4. Based on the data given in Question 3, if you were able to titrate to the third equivalence point of H$_3$PO$_4$, estimate the total volume of NaOH would you need.
5. One source in the literature quotes 0.54 g/L as being the concentration of phosphoric acid in Colas. What is it in molarity? Show your calculations and include units.

**Post-Lab Questions:** (Please type your answers. Equations can be hand-written.)

1. As explained in the previous experiment, the pH of a half-neutralized solution of a weak acid is equal to the pKₐ of the acid. The following questions refer to the titration curve in Figure 10.2 (not your own curve).
   a) What is the volume of NaOH at the first equivalence point?
   b) When half of this volume of NaOH had been added, the solution is half-neutralized. What is the pH at that point?
   c) What is the pKₐ of the acid in the titration?
   d) What is the Kₐ of the acid?

2. Examine Figure 10.2. In the region where the volume of NaOH added was 1 to 9 mL, explain in your own words why the pH is increasing rather than staying constant as it would have been if the acid were HCl. This is the region where the NaOH is reacting with H₃PO₄. Write the net ionic equation to show this reaction. Hint: What are the products in this region of the titration? If the acid were HCl, what would the products have been in this region? How does the difference in products affect the pH in this region?

3. Squirt contains citric acid instead of phosphoric acid. Provide a reason why a Squirt titration is more difficult than a Cola titration. (Hint: Examine the acid dissociation constant provided in the Introduction section.) Answer in full sentences.

4. Why is the first equivalence point at a pH below 7? Explain clearly in full sentences. Use net ionic equations to help illustrate your explanation.
EXPERIMENT 10: TITRATION OF A COLA PRODUCT
Calculations & Results:  
Name: ________________________________

CHEM 124 Sec: _____  
Partner’s Name: ________________________________

Net ionic equation for the titration up to the 1st equivalence point:

Volume of NaOH needed to reach the 1st equivalence point = ________

Calculate the molarity of the phosphoric acid based on the 1st equivalence point:

Net ionic equation for the titration up to the 2nd equivalence point:

Volume of NaOH needed to reach the 2nd equivalence point = ________

Calculate the molarity of the phosphoric acid based on the 2nd equivalence point:

Hint: Do you expect this to be the same or different from your answer for the 1st equivalence point?

SUMMARY OR RESULTS:

<table>
<thead>
<tr>
<th>Molarity of H₃PO₄</th>
<th>Based on 1st equiv pt</th>
<th>Based on 2nd equiv pt</th>
<th>Average</th>
</tr>
</thead>
</table>

Based on the answer to Pre-Lab Question 5, calculate the error and percent error of your average molarity. Show setup here:

ANS. error = ________  
% Error = ________

(continued next page)
Name: ____________________________

Average molarity of $\text{H}_3\text{PO}_4 = \underline{\text{____________}}$ (from previous page)

Calculate the mass percent of $\text{H}_3\text{PO}_4$ in the Cola sample from the average molarity:

ANS. _____________