Name:____ Section____________

Chemistry 118 Laboratory
University of Massachusetts Boston

pH Measurement and Titration

LEARNING GOALS

1. Gain experience using acid/base indicators
2. Learn how to use a pH meter
3. Gain experience with an acid/base titration
4. Gain practice reading a burette

INTRODUCTION

pH is a measure of the acidity of a solution. It is defined as the $-\log[H_3O^+]$. H_3O^+ (protonated water) is the strongest acid that can exist in water. Any stronger acids, such as HCl, completely dissociate in water to form H_3O^+ and an anion (Cl^- in the case of HCl). Weak acids do not completely dissociate when dissolved in water. The measure of the strength of a weak acid is its dissociation constant (K_a); the smaller the acid dissociation constant, the weaker the acid and the smaller its extent of dissociation in water. More basic conditions are required to remove the protons from weaker acids. The pKa, which is defined as the $-\log K_a$, is pH at which the concentration of the weak acid and base conjugate pairs are equal.

Acid-base indicators

An acid-base indicator is a weak acid-base conjugate pair for which the conjugate acid has a different color from the conjugate base. When a small amount of the indicator is added to a solution whose pH is much less than the pK_a of the indicator, the acid form of the indicator predominates, and the solution takes on the acid form's characteristic color. Conversely, when the indicator is placed in a solution whose pH is much greater than the pK_a of the indicator, the base form of the indicator predominates, and the solution takes on the base form's characteristic color. If the solution has a pH near the pK_a of the indicator, then both forms of the indicator are present, and the color of the solution is a mixture of the two indicator colors. For example, bromocresol green (BG) is yellow in solutions more acidic than pH 3.8 and is blue in solutions more basic than pH 5.4. If the solution pH is inside this range, say pH 4.5, the color is a mixture of the acidic yellow and basic blue and is seen as green. The table below lists the six indicators and the abbreviations for them that we will use in this experiment, along with their characteristic colors in high- and low-pH solutions and the transition range for the color change.

Indicator	Color at Low pH	Transition Range	Color at High pH
benzopurpurine (BP)	Violet	2.2-4.2	Red
bromocresol green (BG)	Yellow	3.8-5.4	Blue
methyl red (MR)	Red	4.8-6.0	Yellow
bromothymol blue (BB)	Yellow	6.0-7.6	Blue
thymol blue (TB)	Yellow	8.0-9.2	Blue
Alizarin yellow (AY)	Yellow	10.1-12.0	Orange-Red

Weak acid titration

In this experiment you will titrate H_3PO_4 with a dilute solution of NaOH. H_3PO_4 is phosphoric acid. It is the major acid that gives cola its acidity. It is a triprotic weak acid, which means contains three acidic protons. Therefore, it has three acid dissociation constants; $K_{a1} = 7.11\cdot10^{-3}$, $K_{a2} = 6.34\cdot10^{-8}$ and $K_{a3} = 8\cdot10^{-13}$. The pH of the cola is fairly low, so that much of the phosphoric acid starts out in the H_3PO_4 form. As NaOH is added during the course of the titration, the most acid proton is removed to form $H_2PO_4^-$ and H_2O. At a pH of about 4.6 all of the H_3PO_4 has been converted to $H_2PO_4^-$. This point is called the first equivalence point. If NaOH is added beyond this point, it begins to react with $H_2PO_4^-$ to form HPO_4^{2-}. At a pH of about 9.6 all of the $H_2PO_4^-$ has been converted to HPO_4^{2-}. This point is called the second equivalence point. If NaOH is added beyond this point, it begins to react with HPO_4^{2-} to form PO_4^{3-}. However, HPO_4^{2-} is a very weak acid and the third equivalence point can not be reached in this type of titration. In addition, competition with other ingredients in the cola obscures the chemistry of subsequent neutralization at the second endpoint. Therefore, we will only attempt to determine first equivalence point; the point at which the first hydrogen ion of H_3PO_4 has been neutralized. The net ionic equation for this neutralization is

$$H_3PO_4 + OH^- \rightarrow H_2PO_4^- + H_2O$$

(Note that both H_3PO_4 and $H_2PO_4^-$ are written in molecular form, because they are weak electrolytes.)

A titration curve is a plot of the pH vs. volume of NaOH added. The titration curve for this experiment will start out at a pH near 2, slowly climb in the buffer region where there is a mixture of H_3PO_4 and $H_2PO_4^-$ and take a steep climb as the equivalence point is reached. Beyond the equivalence point the curve will flatten once again as it enters the second buffer region where there is a mixture of $H_2PO_4^-$ and HPO_4^{2-}. This is the point where we will stop our titration. The volume it takes to reach the equivalence point can be used to find the concentration of the phosphoric acid in the cola, because an equivalent number of mmoles of NaOH is required to completely convert all of the H_3PO_4 to H_2PO_4-. The number of mmoles of NaOH used to reach the first equivalence point is V_{eq}·[NaOH], where V_{eq} (in mL) is the volume of NaOH required to reach the equivalence point and [NaOH] is the concentration of the titrant in mol/L or (mmol/mL). Therefore, the concentration of the phosphoric acid in the cola is given by

$[H_3PO_4] = V_{eq} \cdot [NaOH] / V_{cola}$

where V_{cola} is the volume of the cola that was analyzed (in mL).

We will use a pH meter to measure the pH after the addition of successive, small increments of NaOH from a buret. A pH meter is an electronic instrument that measures the pH of a solution by detecting the electrical potential between a reference electrode and a special indicating electrode with a thin glass bulb at its end. Most pH meters today use a combination electrode, which brings together the reference and indicating electrodes in a single, glass housing. The tip of the electrode is usually protected with a plastic guard to prevent breakage of the delicate indicating glass membrane. Because there may be more than one model of pH meter in the laboratory, your instructor will give instructions about the operation of the particular meter you may be using.

PROCEDURE

Part 1 Using Acid-Base Indicators to Estimate the pH of a solution

In this part you will use these six indicators to find the approximate pH range of two unknown solutions. Your objective is to determine the pH within the narrowest possible range. This means that you must determine the pH of each unknown solution between the transition ranges of two adjacent indicators or within the range of a single indicator. For example, suppose your unknown solution turns bromothymol blue (BB) to blue and thymol blue (TB) to yellow. The blue BB color indicates that the unknown solution has a pH greater than 7.6, and the yellow TB color indicates that it has a pH less than 8.0. Together these two observations narrow down the unknown solution's pH to the range 7.6 – 8.0. This is the narrowest range that can be detected for the unknown solution with this set of indicators. Of course, it would be correct to claim that the unknown solution's pH is within a wider range, say 2.2 – 12.0, but we are looking for the narrowest range. You will be assigned two different unknowns. Write the two codes for the unknowns in the spaces provided in the first column of the data table on page 6. Then carry out the following procedures on each unknown solution, one at a time.

- □ Obtain 18 small disposable test tubes. Arrange these in the test tube rack in three rows of six each. Fill each of the test tubes in the top row with 10 drops of 1 M NaOH, to a level of about 1 cm. In the bottom row, fill the first four tests tubes (left to right) with 10 drops of 1M HCl, and fill the last two test tubes with the same amount of deionized water (see the data table on p. 7). Each of the six test tubes in the middle row should be filled with 10 drops of the unknown solution you are testing.

- □ Add 2 drops of the indicator, as specified on the data table, to the three test tubes in a column on your test tube rack (higher-pH reference, unknown, lower-pH reference). Be certain that you know or mark the contents of each test tube in the rack.

□ Check that
  - each top row solution is the proper color for the indicator above its transition range;
  - each bottom row solution is the proper color for the indicator below its transition range.

□ In the appropriate boxes of the data table, record the colors of the six test tubes containing the unknown. Compare the unknown's colors with the colors of each indicator in its higher-pH form (top row) and its lower-pH form (bottom row). Use these comparisons to deduce the pH of the unknown solution within the narrowest possible range. Write your conclusions in the spaces provided below the data table.

□ After you are finished with your first unknown, empty the contents of the six sample test tubes (middle row) in the designated waste container, and discard the empty test tubes in the glass disposal receptacle. Obtain six new test tubes for your second unknown sample, and proceed in the same manner as you did with the first sample.

Part 1 Titration of cola

□ Obtain a 200- or 250-mL beaker to serve as a temporary liquid waste receptacle. You will empty the contents of this beaker into the designated laboratory waste container at the end of the experiment.

□ In another *clean and dry* beaker, obtain approximately 75 mL of 0.025 M NaOH solution. Use a little of this to rinse a 50-mL buret, discarding the wash liquid into your temporary waste beaker.

□ Fill the buret above the 0-mL line, then open the petcock over your temporary waste beaker to expel any air bubbles in the tip. Adjust the level in the buret to the 0-mL line. This will simplify the data collection and calculations.

□ Rinse a clean 200- or 250-mL beaker with deionized water. Measure 50.0 mL of cola and 50.0 mL of deionized water, using a graduated cylinder. Transfer both liquids to the beaker.

□ Set the beaker in the center of a magnetic stirrer, and carefully drop in the magnetic stir bar. Turn on the stirrer's motor and adjust the speed for a slow and steady rotation.

□ The combination electrode of the pH meter should be soaking in a pH 7.00 buffer solution. If the reading is more than 0.05 pH units from 7.00, ask your instructor to check the calibration and function of the pH meter.

□ Remove the electrode from the buffer solution and rinse it in a stream of deionized water from a wash bottle, collecting the wash liquid in your temporary waste beaker. Insert the electrode into the cola sample solution. Check to be sure the tip of the electrode is just far enough from the bottom of the beaker to avoid being hit by the spinning stir bar.

□ Record the initial solution pH and all subsequent pH readings on the data table on p. 8. Titrate by adding 1.0-mL portions of the NaOH solution. It is not critical that each addition be exactly 1.0 mL, ***but record what your actual volume was at each point, reading the buret to ±0.01 mL***. After each addition record the measured pH on the data table. Continue the titration until you have added at least 6 mL beyond the inflection point volume.

- □ Clean up as follows:
  - ○ Rinse the electrode with deionized water, and return it to the buffer solution in which it was originally soaking.
  - ○ Leave the stir bar on top of the magnetic stirrer.
  - ○ Empty the buret into your temporary waste beaker. Rinse out the buret thoroughly with deionized water, and leave it in an inverted position in the buret clamp to allow complete draining.
  - ○ Empty your temporary waste beaker and any leftover stock NaOH solution into the designated laboratory waste container.
  - ○ Rinse and return all glassware.

pH Measurement and Titration

Name____ Section_______________________

Partner(s)____

Part A: Determining Approximate pH of Unknown Solutions

higher pH solutions	NaOH + BP pH ≥ 4.2 red	NaOH + BG pH ≥ 5.4 blue	NaOH + MR pH ≥ 6.0 yellow	NaOH + BB pH ≥ 7.6 blue	NaOH + TB pH ≥ 9.2 blue	NaOH + AY pH ≥ 12.0 orange-red
unknown #1 Code:						
unknown #2 Code:						
lower pH solutions	HCl + BP pH ≤ 2.2 violet	HCl + BG pH ≤ 3.8 yellow	HCl + MR pH ≤ 4.8 red	HCl + BB pH ≤ 6.0 yellow	H_2O + TB pH ≤ 8.0 yellow	H_2O + AY pH ≤ 10.1 yellow

Conclusions: The pH of Solution #1, Code _______, is great than ______ but less than ______.

The pH of Solution #2, Code _______, is great than ______ but less than ______.

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Part B: Titration of Phosphoric Acid in Cola

Target Volume (mL)	Actual Volume (mL)	pH	Target Volume (mL)	Actual Volume (mL)	pH
0			13		
1			14		
2			15		
3			16		
4			17		
5			18		
6			19		
7			20		
8			21		
9			22		
10			23		
11			24		
12			25		

Endpoint volume from pH vs. NaOH volume plot ______________

Millimoles of NaOH added at endpoint = millimoles H_3PO_4 in sample ______________

Mass of H_3PO_4 in 50-mL sample of cola ______________

Mass of H_3PO_4 in a 2.0-L bottle of cola ______________

Lab Report

No abstract required

The lab report will consist of your data sheet and the Part 1 and 2 described below. The data sheet is worth 30 points

Part 1:
Describe the observations made for each unknown (4 pts for each unknown) and discuss how these observations enabled you to narrow the window for the possible pH values of your unknowns (4 points for each unknown).

Part 2:
Construct a titration plot using Excel. Label the axes and title the plot. (7 pts) Report the equivalence point volume and the concentration of H_3PO_4 in (mol/L) in your cola sample as calculated from the equivalence point volume. (7 points)