Experiment 29: pH Measurements and Its Applications
(This experiment was adapted from Santa Monica College.)

Purpose: The measurement of pH will be practiced and applied to solution questions.
   a) The pH of three different solutions will be estimated with pH paper and measured with a pH meter.
   b) An unknown acid solution will be used to prepare a series of solutions by serial dilution, and the pH of each solution will be measured with a pH meter. Knowing the concentration and pH of each solution will allow for the calculation of the Ka for this unknown acid.
   c) A buffered solution will be prepared with a weak base and its conjugate acid, Na_2CO_3 and $NaHCO_3$. The behavior of this buffer will be observed when a strong acid and a strong base is added. This buffer behavior will be compared to the pH changes that occur when strong acid and strong base are added to deionized water.

Background: The pH of a solution is a value that represents the concentration of H_3O^{1+} in that solution. It is an easy-to-convey value, rather than stating concentrations in scientific notation. The most common pH scale contains values from 0 to 14. pH values less than 7 are for acidic solutions. pH values greater than 7 are for basic solutions. A pH value equal to 7 is for neutral solutions.

The pH value of a solution is calculated with the following equation:
   pH = - log [H_3O^{1+}]

The pH of a solution can be estimated with pH paper; that is paper impregnated with indicators. Indicators are chemicals that have a color that depends on the pH of their environment. The value you get with pH paper is an estimated value; no significant figures.

The pH of a solution can be measured with a pH meter. The pH meter has an electrode that is sensitive to the concentration of H_3O^{1+}. The pH meter will report out pH values with two significant figures. Commercial buffer solutions are used to check the accuracy of the pH meter.

Once the pH of a solution is known, the concentration of H_3O^{1+} can be calculated. The following equation is used for this:
   [H_3O^{1+}] = 10 $^{-pH}$

A weak acid (HA) dissolved in water reacts with the water as follows:
   $$HA + H_2O \rightleftharpoons A^- + H_3O^+$$

The Ka of a weak acid is calculated with the following equation:
   $$Ka = ([A^-] [H_3O^+]) / [HA]$$
The pH of the solution can be used to calculate the [H_3O^+], which is equal to the [A^-] (both are equilibrium values). The [HA] equilibrium value can be calculated as (initial – used = equilibrium).

A buffered solution is a solution that contains a weak acid and its conjugate base, or a weak base and its conjugate acid. The pH of a buffer can be calculated with the following equation:

pH = pKa + log ([buffer base] / [buffer acid]),   and pKa = - log Ka

The pH of a buffer does not change very much when an acid or base is added to the buffer. The buffer base will neutralize incoming acid and the buffer acid will neutralize incoming base.

Chemicals and Equipment:
$NaCl_{(s)}$ $NaHCO_{3(s)}$ $Na_2CO_{3(s)}$ 0.10 M unknown acid   6 M HCl   6M NaOH
pH paper   pH meter   pipets, 100 mL volumetric flasks   assorted beakers

Procedure:
Part A: Prepare three different solutions, each with a concentration of 0.010 M.
  1) Obtain solid NaCl from the stock bottle and put it into a clean, dry beaker. You will only need about 1 scoop of this solid.
  2) Use tared weighing paper at the lab balance, and measure out the appropriate amount of solid NaCl to make 100 mL of a 0.010 M solution. Record this mass of NaCl in your notebook.
     (calculate moles and then grams, M=mole/L, mole x g/mole = g)
  3) Transfer the solid NaCl from the weighing paper into a 100 mL volumetric flask.
  4) Add deionized water to the flask to dissolve the solid. Then continue to add the water to dilute to the graduation mark on the neck of the flask. Make sure the bottom of the meniscus sits exactly at the calibration mark.
  5) Cover the flask with Parafilm, secure with your thumb, and invert to mix well (30 – 50 inversions). Save this solution.
  6) Repeat steps 1 – 5 with $NaHCO_3$.
  7) Repeat steps 1 – 5 with Na_2CO_3.
  8) Put approximately 30 mL of each of the three solutions into its own clean, dry beaker.
  9) Use a clean, dry glass stirring rod to take a drop of solution from the beaker and put it onto a piece of pH paper. Estimate the pH of each solution using the color chart supplied with the pH paper. Record these pH values in your notebook.
  10) Use the pH meter to measure the pH of each solution. Record these pH values in your notebook.

In the data section of your notebook, tabulate the molarity concentration of each solution, the ions present in solution, the pH obtained with pH paper, and the pH obtained with the pH meter.
In the calculation/results section of your notebook, mention what you expected the pH to be (pH <7, =7, >7) based on the ions in solution (were the ions weak acids, just spectators, or weak bases). Show the calculations for the molarity of each solution.

Part B: Calculation of the Ka for an Unknown Weak Acid
    Perform serial dilutions to prepare several solutions of different concentrations.
1) Obtain 20 mL of the 0.10 M unknown acid from the stock bottle in the fume hood.
2) Pipet 10.0 mL of this acid into a 100 mL volumetric flask.
3) Add deionized water to the flask to dilute the acid to the calibration mark. Make sure the bottom of the meniscus sits exactly at the calibration mark.
4) Cover the flask with Parafilm, secure with your thumb, and invert to mix well (invert 30 – 50 times). This is solution B1.
5) Pipet 10.0 mL of this new solution, B1, into a new 100 mL volumetric flask and dilute to volume, following steps 3 – 4. This is solution B2.
6) Pipet 10.0 mL of this new solution, B2, into a new 100 mL volumetric flask and dilute to volume, following steps 3 – 4. This is solution B3.
7) Pipet 10.0 mL of this new solution, B3, into a new 100 mL volumetric flask and dilute to volume, following steps 3 – 4. This is solution B4.
8) Calculate the concentration of unknown acid in each of these solutions (B1, B2, B3, and B4). (This is the initial concentration, [HA], for the Ka equation.)
9) Measure the pH of each of these solutions.
10) Use the measured pH values to calculate the equilibrium $[H_3O^+]$ in each solution.

In the data section of your notebook, tabulate the molarity of weak acid in each solution (this will be the initial concentrations), and the measured pH values.

In the calculation/results section of your notebook, compare the calculated initial concentration of weak acid in each solution with the equilibrium concentration of H_3O^+ in each solution (calculated with $[H_3O^+] = 10^{-pH}$). Does the weak acid ionize 100%? Calculate the value of Ka for each solution. Is it ever okay to assume that the equilibrium concentration of HA is equal to the initial concentration of HA in each solution when doing the calculation of Ka?

Part C: Preparation of a Buffered Solution
Prepare a buffered solution (referred to as the original buffer).
1) Weigh 0.840 g of $NaHCO_3$ into a 100 mL beaker (tare the beaker).
2) Weigh 1.060 g of Na_2CO_3 into the same beaker (tare the beaker + $NaHCO_3$).
3) Add approximately 50 mL of deionized water to the beaker, and stir with a glass stirring rod until the solid is dissolved.
4) Rinse the glass stirring rod off with deionized water, while holding it above the beaker. This is to make sure you don't lose any of the dissolved solutes.
5) Transfer the solution into a 100 mL volumetric flask, making sure not to lose any solution. Rinse the beaker with three 10 mL portions of deionized water, transferring each rinse into the volumetric flask.
6) Dilute the solution to the graduation mark on the flask, making sure that the bottom of the meniscus sits exactly at the calibration mark.
7) Cover the flask with Parafilm, secure with your thumb, and invert to mix well (invert 30 – 50 times).

Now the original buffer solution is prepared. Before using the buffer, divide it into two equal portions. Do this by pouring 50.0 mL of the buffer into a graduated cylinder. Then transfer each 50 mL portion of buffer into its own clean, dry 100 mL beaker (buffer 1 and buffer 2). You will be observing how the pH of a buffer does not change much when an acid or base is added. You will also be observing what happens when the buffer capacity is exceeded (when one of the buffer components is used up and can no longer do its buffer job).

8) Use the pH meter to measure the pH of one of these buffer portions (since both portions are the same, the pH will be the same for both).
9) Add 0.50 mL of 6.0 M HCl to buffer 1; mix. Measure the pH with the pH meter.
10) Add an additional 0.50 mL of 6.0 M HCl to buffer 1; mix. Measure the pH with the pH meter.
11) Add 0.50 mL of 6.0 M NaOH to buffer 2; mix. Measure the pH with the pH meter.
12) Add an additional 0.50 mL of 6.0 M NaOH to buffer 2; mix. Measure the pH with the pH meter.
13) Use a new, clean, dry 100 mL beaker. Add 50.0 mL of deionized water to the beaker. Measure the pH of this water with the pH meter. The pH may not be exactly 7.
14) Add 0.50 mL of 6.0 M HCl to this water sample; mix. Measure the pH with the pH meter.
15) Use a new, clean, dry 100 mL beaker. Add 50.0 mL of deionized water to the beaker. Assume the pH is the same as in step 13.
16) Add 0.50 mL of 6.0 M NaOH to this water sample; mix. Measure the pH with the pH meter.

For part C, in the data section of your notebook, tabulate all of the pH values you measured.

In the results section of your notebook, show the following:
a) Calculate the molarity of each buffer component. (g to moles, moles / L = M)
b) Calculate the expected pH of the original buffer solution.
   (pH = pKa + log([b.base]/[b. acid]))
c) Calculate the molarity of the added HCl and added NaOH.
   (Mc*Vc=Md*Vd, solve for Md)
d) Write the neutralization reaction for the original buffer when HCl is added.
e) Calculate the new buffer molarities after the first addition of HCl (assume no dilution of the buffer components, but take into account the neutralization).
f) Calculate the new buffer pH after the first addition of HCl.
g) Write the neutralization reaction for the original buffer when NaOH is added.
h) Calculate the new buffer molarities after the first addition of NaOH (assume no dilution of the buffer components, but take into account the neutralization).
i) Calculate the new buffer pH after the first addition of NaOH.
j) Explain the sudden change in pH after the second addition of HCl and NaOH were added.

In the conclusion section of your notebook, comment on the results of each part of this experiment with regard to each purpose:

In Part A, you obtained pH values using pH paper and a pH meter. Compare and comment on these results. You prepared three different solutions; how did the pH values compare? Were they as you expected given the ions that existed in solution? Explain why.

In Part B, you prepared several solutions of a weak acid by serial dilution. How did the pH results compare relative to the initial concentration of weak acid in each solution? You calculated the Ka of the weak acid, in each solution. How did the Ka values compare? Was Ka constant? Were you able to assume that $[HA]_{initial}$ could be used in the calculation of Ka, instead of $[HA]_{equilibrium}$?

In Part C, you prepared a buffered solution. This was a weak base with its conjugate acid buffer. Knowing this, is the pH of the original buffer what you expected? When you added the first portion of HCl and of NaOH, did the pH change much? Why or why not? What happened when the second portion of HCl and of NaOH was added? Why did the pH change drastically then? (Refer to buffer capacity when you answer this). Comment on the drastic change in pH of the water samples when HCl was added, and when NaOH was added.