EXPERIMENT 11
THE MOLAR MASS OF A WEAK DIPROTIC ACID

PURPOSE:

To determine the Molar Mass of an unknown diprotic acid, by an acid-base titration.

PRINCIPLES:

This experiment is a continuation of a previous experiment (Experiment # 9B) and is based on the same general principles of an acid-base titration. An indicator is used to detect the end point of the titration. The "End Point" is the point in the titration when the indicator changes color. Since the indicator used for this titration is correctly chosen (phenolphthalein) the "End Point" occurs very close to the "Equivalence Point" (the point in the titration when the number of moles of acid used equals exactly the number of moles of base added).

Also, this experiment follows a reverse scenario: the molarity of the NaOH solution used for the titration is known, whereas the molar mass of the unknown weak acid is unknown. Furthermore, the stoichiometry of the acid-base neutralization is more complex since the acid and the base release an unequal number of H_3O^+ and OH^- ions (this will be discussed below)

The unknown weak diprotic acid, whose molar mass is to be determined in this experiment, is a soluble solid, containing two acidic hydrogen atoms and is commonly and is abbreviated as H_2A. When dissolved in water, the solid weak diprotic acid undergoes partial ionization in two successive steps, according to the following acid ionization equilibria:

Step 1: $H_2A\,(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + HA^-(aq)$

Step 2: $HA^-\,(aq) + H_2O(l) \rightleftharpoons H_3O^+(aq) + A^{2-}(aq)$

As it is the case for all the Acid Ionization Equilibria of Week Acids, both these equilibria proceed to a very low extent in the forward direction. This is so because in the first step the first proton (H^+) separates from a neutral molecule (H_2A). In the second step the second proton (H^+) must separate from a negatively charged anion (HA^-) which holds the H^+ more tightly than the neutral H_2A molecule. This means that the solution contains:

1. mostly intact (or un-ionized) H_2A molecules
2. a few HA^- ions, and
3. even fewer A^{2-} ions

In order to determine the molar mass of an unknown diprotic acid (g/moles) the number of moles of H_2A that correspond to a known mass of H_2A needs to be determined experimentally. The number of moles H_2A in an aqueous solution can be determined if the weak acid is forced into complete ionization in the above two steps. The complete ionization of the weak acid H_2A can be achieved by successive additions of a standardized solution of sodium hydroxide. The addition of NaOH to this equilibrium system will cause the OH^- ions to combine with the hydronium (H_3O^+) ions produced by the weak acid to form water:

$$OH^-(aq) \quad + \quad H_3O^+(aq) \longrightarrow 2H_2O(l)$$

This decreases the concentration of hydronium (H_3O^+) ions and causes the equilibrium system of the first ionization step to shift to the right, producing the weak acid (HA^-) which will start to ionize in the second ionization step. As a result, both ionization equilibria of the weak acid (first and second ionization steps) will shift to the right and force the weak acid into complete ionization.

The situation can be summarized in the equilibrium tables below:

First Ionization Step

	H_2A (aq)	+	H_2O(l)	$\rightleftharpoons$	H_3O^+(aq)	+	HA^-(aq)
Stress:					decreases		
Shift:				$\Rightarrow$			
New Equilibrium:	decreased		decreased		decreased		increased

Second Ionization Step

	HA^- (aq)	+	H_2O(l)	$\rightleftharpoons$	H_3O^+(aq)	+	A^{2-}(aq)
Stress:					decreases		
Shift:				$\Rightarrow$			
New Equilibrium:	decreased		decreased		decreased		increased

Overall

	H_2A (aq)	+	$2H_2O$(l)	$\rightleftharpoons$	$2H_3O^+$(aq)	+	A^{2-}(aq)
Stress:					decreases		
Shift:							
New Equilibrium:	decreased		decreased		decreased		increased

The net result is the complete neutralization of the weak diprotic acid (H_2A) by the NaOH solution:

Step 1:	H_2A(aq)	+	NaOH(aq)	$\longrightarrow$	~~NaHA~~(aq)	+	H_2O(l)
Step 2:	~~NaHA~~(aq)	+	NaOH(aq)	$\longrightarrow$	Na_2A(aq)	+	H_2O(l)
Overall:	H_2A(aq)	+	2 NaOH(aq)	$\longrightarrow$	Na_2A(aq)	+	$2H_2O$(l)
Net Ionic Equation:	H_2A (aq)	+	$2OH^-$(aq)	$\longrightarrow$	A^{2-}(aq)	+	$2H_2O$(l)

EXPERIMENT 11
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Since the weak acid is forced into complete ionization, the concentration of the [H_3O^+] ions produced by the weak acid is related to the number of moles of H_2A. The stoichiometry of the reaction indicates that 2 moles of H_3O^+ ions are produced for every mole of completely ionized H_2A. The concentration of [H_3O^+] can be determined experimentally since it will combine with two moles of OH^- ions provided by the standardized solution of NaOH during an acid-base titration. In brief, performing an acid-base titration allows the concentration of the [H_3O^+] to be determined by titrating an aqueous solution of H_2A, containing a known mass of the weak acid, with a NaOH solution of a known molarity.

As noted above, twice as many moles of NaOH are needed than the moles of the weak acid.

$$\boxed{\textbf{Number of moles of NaOH} = \frac{\textbf{Number of moles of } \mathbf{H_2A}}{\mathbf{2}}}$$

PROCEDURE:

1. For the first titration, weigh out between 0.10g and 0.15g of the unknown weak acid into a 250 mL Erlenmeyer flask, using the weighing bottle technique.

   a. This mass may need to be adjusted for the subsequent titrations, depending on the unknown weak acid assigned to you.

   b. In order to obtain accurate experimental data, save time and reagent, it is suggested that the volume of NaOH used to reach the end point should be between 15mL and 30mL.
   The rationale for this is as follows:
   - The volume of titer used should not be less than 10 mL.
     Using less than 10 mL of titer to reach the end point would result in a final of the Molar Mass in three significant figures only.
   - The volume of titer used should not be more than 30 mL
     - This ensures that sufficient NaOH solution is available for all required titrations, and
     - This allows for an efficient use of time (less volume delivered used to reach the end point will take less time)

EXPERIMENT 11
THE MOLAR MASS OF A WEAK DIPROTIC ACID

2. Guidelines for adjusting the mass of the weak acid weighed out:
   Example 1: TOO LITTLE
   Assume that for the first titration you weighed out 0.1835g of the acid, and 8.45mL of NaOH was used to reach the end point. In this case, the volume of NaOH is too small (3 significant figures), and more mass of the acid would need to be weighted out for subsequent titrations in order to use more NaOH. As a rule of thumb, use about 20mL NaOH per titration. You can estimate the range of the mass of acid you need to weigh out:

$$\frac{0.1835\text{g acid}}{8.45\text{mL base}} \quad \frac{\text{Xg acid}}{20\text{mL base}} \qquad X = \frac{(0.1835\text{g})\ (20\text{mL})}{8.45\text{mL}} \approx 0.43\text{g}$$

   In this example, for the subsequent titrations, about 0.5g of acid is needed.

   Example 2: TOO MUCH
   If your first titration required too much NaOH to reach the end point (more than 40 mL) decrease the mass of acid to be weighed out for future titrations. Use the same estimation calculation shown in the example above to estimate the mass of acid that would require about 25mL of NaOH to reach the end point.

3. Perform at least five titrations (very often the first titration, and/or the second will be in error).
   a. Your aim is to obtain an accurate mean value for the molar mass.
   b. This experiment is graded on sliding scale of percent error comparing the reported experimental value to the theoretical value.
4. Evaluate your data by calculating the standard deviation.
   a. At least more than half of your titrations must be included in the calculation of the mean molar mass.
   b. If the standard deviation is not within an acceptable range you may discard the titrations in error and perform additional titrations.
   c. You are not required to turn in the standard deviation calculations for a grade, but you are required to report the standard deviation "s" on the report form.
5. Clean-up instructions:
   a. Discard the titration waste into the sink
   b. DO NOT DISCARD THE UNUSED STANDARDIZED SOLUTION OF NaOH!
      - It will be used in the next experiment!
      - Use a stopper to firmly seal the bottle containing the NaOH solution and store it in your locker.

EXPERIMENT 11
THE MOLAR MASS OF A WEAK DIPROTIC ACID

REPORT FORM

NAME: _________________________ UNKNOWN #: ____________ Date: ____________

Molarity of standardized NaOH solution: ____________ M

	1st Titration	2nd Titration	3rd Titration	4th Titration	5th Titration
Initial mass of H_2A + vial (g)					
Final mass of H_2A + vial (g)					
Mass of H_2A used (g)					
Final buret reading (mL)					
Initial buret reading (mL)					
ML of NaOH used					
L of NaOH used					
Moles of NaOH used					
Moles of H_2A used					
Mass of H2A used (g)					
Molar mass of H_2A (g/mol)					
Standard Deviation “s”					
Mean molar mass (g/mol)					

The Mean Value of the Molar Mass of the
Diprotic Weak Acid probably lies between __________ ± __________ (g/mole)

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