EXPERIMENT 5
IDENTIFICATION OF A POLYPROTIC ACID VIA TITRATION

A. Prelab

- Read Skoog et al. sections 13A-B (pp. 303-305) and 13-D (pp. 308-314) for background information.
- You have one week to complete the experimental portion of the lab.

B. Experimental Overview

This week you will perform a titration of a polyprotic acid with your standardized NaOH solution using a pH meter as an indicator of solution pH. You will work in groups of three or four and share data. The primary purpose of this titration is to accurately map out the titration curve of a weak polyprotic acid with a strong base. You will then determine the position of the equivalence points of the reaction graphically using a titration curve, a first derivative titration curve, and a second derivative titration curve. You will then determine two or more pK_a values and use these values to identify your unknown based on comparison with literature values.

C. Procedure

Here is a general procedure for a titration with a pH meter:

- Calibrate the pH meter according to the pH meter manual. You will calibrate your pH meter with at least 3 pH buffers. The meter will automatically perform a linear regression for you.

- Prepare your notebook. You will record pH vs. mL base added as you titrate. You will also need to prepare a full-page graph on provided graph paper that will cover the pH and mL ranges before you begin. You will need to record data and plot pH vs. mL base added as you titrate.

- Condition and fill a 50 mL buret with standardized 0.1 M NaOH solution. Record its concentration in your notebook.

- Pipet 1 mL of your unknown into a 100 mL volumetric flask and dilute to the line. Transfer all 100 mL of this solution into a 250 mL beaker for titration.

- Titrate your solutions by adding larger aliquots of base (1 mL) during buffer regions, where the pH does not change rapidly and smaller amounts of base (0.1 to 0.5 mL) near the equivalence points.

<u>Data Analysis</u>

- Plot your data as pH vs. volume of base added (mL). This is a titration curve.
- Plot the first derivative of pH vs. Volume of base added (mL). This is a first derivative titration curve.
- Plot the second derivative of pH vs. volume of base added (mL). This is a second derivative titration curve.
- Identify the equivalence points from your all three of your titration curve plots.
- Calculate the number of moles of NaOH titrated at each equivalence point.

- Determine the pK_as for your unknown. (You can find the first pKa by finding the pH of the solution half-way to the first equivalence point. Additional pK_as exist half-way between higher equivalence points.)
- Identify the best possible candidate from the following list of polyprotic weak acids.

Weak Acids, Ka, and pKa values

Acid	**HA**	**A-**	**Ka**	**pKa**
Carbonic	H_2CO_3	HCO_3^-	4.3 x 10-7	6.37
HCO_3^-	CO_3^{2-}	4.8 x 10-11	10.32	
Citric	$C_6O_7H_8$	$C_6O_7H_7^-$	7.41 x 10-4	3.13
$C_6O_7H_7^-$	$C_6O_7H_6^{2-}$	1.74 x 10-5	4.76	
$C_6O_7H_6^{2-}$	$C_6O_7H_5^{3-}$	3.98 x 10-7	6.40	
Phosphoric	H_3PO_4	$H_2PO_4^-$	7.52 x 10-3	2.12
$H_2PO_4^-$	HPO_4^{2-}	6.23 x 10-8	7.21	
HPO_4^{2-}	PO_4^{3-}	2.2 x 10-13	12.67	

D. Lab Report

- Title Page (include lab title, your name, your section, and unknown #)
- Purpose (be brief, 1 paragraph)
- Data Table
- Four titration curves: a hand plotted titration curve, a computer plotted titration curve, a first derivative titration curve, and a second derivative titration curve. **Identify** and **label** the **equivalence points** and **pK_as** *on each plot*.
- Equations: describe how you calculated the first derivative titration data, and the second derivative titration data from your raw data.
- Result: what is the identity of your unknown.

This report will be due in one week.