

MOHAWK

COLLEGE OF APPLIED ARTS AND TECHNOLOGY

**CHEMICAL, ENVIRONMENTAL, AND BIOTECHNOLOGY
DEPARTMENT**

Calibration of a pH Meter and Gravimetric Titrations

by Professor David Cash

September, 2008

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This Experiment is a 3 hour Analytical Chemistry laboratory exercise. It is designed for students in a common second term course of a 2-year diploma program (Biotechnology, Environmental, or Health Technician).

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Experiment 7 The pH Meter: Calibration of a pH Meter and Titration Curve Applications
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OBJECTIVE

A pH meter will be calibrated and used in two gravimetric acid-base (pH) titrations. The comprehension and skills learned will be transferable to other laboratory and workplace situations.

- Two required NBS¹ standard buffer solutions will be prepared and used to calibrate a digital pH meter.
- Two gravimetric acid-base (pH) titrations will be performed and the titration curves of pH versus titrant mass will be plotted.
- Titration end-point (equivalence) quantities will be estimated from the plotted data.

REFERENCE

Harris

Chapter 6, Problem 6-10, page 127	Chapter 8, pages 153-170	Chapter 9, pages 175-187
Chapter 10, pages 193-212	Chapter 11, pages 231-234 (See Figure 11-3, page 233)	Chapter 15, pages 319-321

INTRODUCTION

(Some of the following is adapted from an Introduction written by Professor Jim Barr, Mohawk College, Retired.)

The pH Meter

The pH meter is one of the commonest instruments used in any laboratory. The correct set-up and use of the instrument and the calibration of the electrode system will be reinforced in this laboratory procedure. The pH meter will be used to perform two gravimetric titrations, for which the titration curves of pH versus titrant mass will be plotted.

In aqueous solution, **pH** is defined as:

$$\text{pH} = -\log [\text{H}_3\text{O}^+]$$

It is determined by the self-ionization of water:



and by the addition of acidic or basic materials to water.

The **pOH** of an aqueous solution is similarly defined as:

$$\text{pOH} = -\log [\text{OH}^-]$$

For the same sample, **pH** and **pOH** are related

by the relationship:

$$\text{pH} + \text{pOH} = \text{pK}_w$$

This Section Continues on the Next Page →

¹ NBS: The United States National Bureau of Standards

The pH Meter (Cont.)

The quantity pK_w is the negative logarithm of the self-ionization constant of pure water, and at ordinary temperatures has a value of approximately 14.

The self-ionization of pure water, however, is temperature dependent, and the “neutral” pH of 7 is strictly true only at $25\text{ }^\circ\text{C}$ when $pK_w = 1.00 \times 10^{-14}$.

The pH of acidic solutions is determined by the added acid, but the pH of basic solutions is determined by the concentration of base and the value of K_w .

For a basic solution: $pH = pK_w - pOH$

or

$$[H_3O^+] = \frac{K_w}{[OH^-]}$$

The pH of a solution can be measured by using a pH meter (a high-resistance voltmeter) and a glass electrode (**Figure 1**).

The diagram shows a combination electrode, with a second electrode, a reference electrode, built in. The two electrodes are sometimes separated.

The membrane is a special glass that allows penetration by water and by hydrogen ions. When the hydrogen ions enter the membrane, a positive voltage is developed because of the positive charge on the ions.

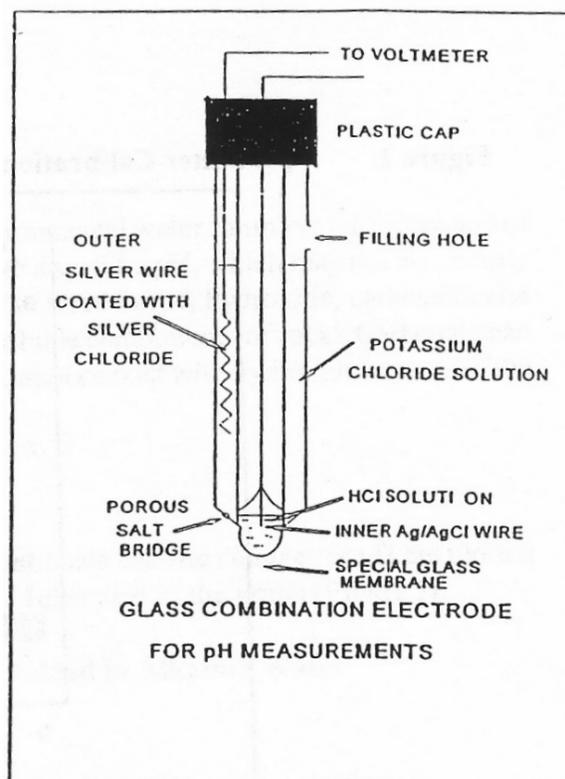


Figure 1: Glass Combination Electrode

By an equilibrium process between the glass and the solution, the potential difference increases when the hydrogen-ion concentration in the solution increases. The voltage is measured against the reference electrode of a fixed electrical potential, in this case a silver wire coated with silver chloride.

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The pH Meter (Cont.)

The voltage or electrode potential, E , of this glass electrode system is given by the relationship:

$$E = \text{constant} - (S \times \text{pH})$$

where the **constant** is specific to a given glass electrode and **S** is the **Nernst Slope**, which has a value of approximately **59 mV** per pH unit but varies with temperature.

The pH meter measures the voltage and converts it to a read-out in pH units directly, with the pH scale marks separated by the theoretical Nernst slope value of **59.1 mV** at **25 °C**.

On most modern pH meters, the readout is digital, but it is still based on the 59 mV interval.

A set of electrodes will give a voltage reading that depends not only on the pH of the sample, but also on the constant in the above equation and on the exact value of the Nernst slope. Since these quantities vary from electrode to electrode, each set must be calibrated with solutions of known pH values, known as pH buffer solutions. The meter will display the correct pH value of the sample only if the electrode response follows the line shown in **Figure 2** below.

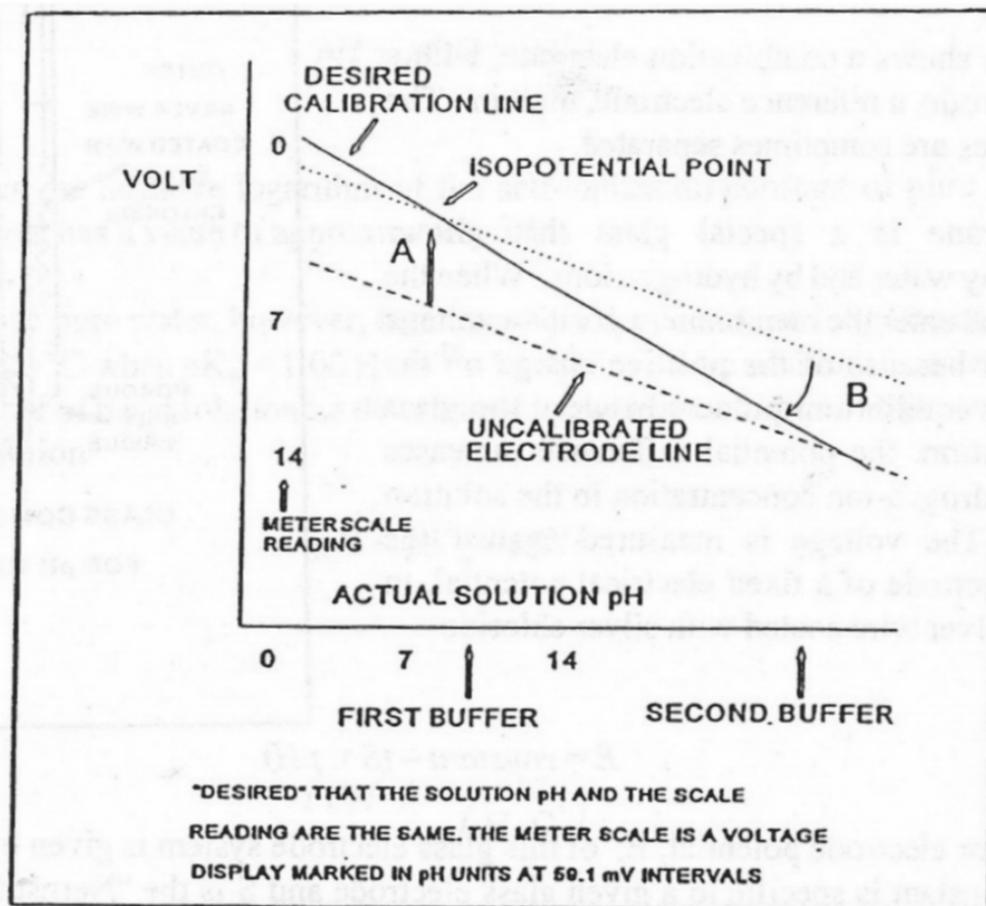


Figure 2: pH Meter Calibration

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The pH Meter (Cont.)

Electrodes that are not calibrated will have an incorrect slope and an incorrect intercept, shown by the dotted line **A** in **Figure 2** on page 3. The process of a calibration consists of adjusting the electrode response line to the ideal line, in two steps:

1. The electrodes are placed in the first buffer solution, which has a specific known pH value. The calibrate control on the meter is adjusted to make the display read the pH of the buffer solution. This process corresponds to the movement of the dotted line **A** shown in **Figure 2** on page 3 toward position **B**. In older pH meters, the adjustment is manual, but it is frequently done by a microprocessor on modern pH meters.
2. The electrodes are rinsed and placed in a second buffer, which has a different specific known pH value. The slope control is then adjusted so that the meter reads the pH value of the second buffer; a correct response line is obtained. Again, the meter may do this adjustment by itself. The meter may also have a separate temperature control, which is set to the sample temperature. Any differences in temperatures during a set of measurements are compensated by adjusting the slope line. The best systems, however, have a temperature sensing probe that reads the sample temperature and automatically adjusts the slope while the measurements are being done.

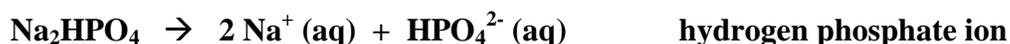
Buffers

A buffer solution is a solution containing a substance or a mixture of substances capable of opposing a change in pH due to the addition of either an acidic substance or a basic substance to the solution. There are several kinds of buffer substances and mixtures. The two NBS buffer solutions used to calibrate a pH meter in this experiment represent two different kinds of buffers.

Phosphate Buffer (d1 Buffer)

The NBS d1 buffer contains an equimolar mixture of **dihydrogen phosphate ion** and **hydrogen phosphate ion**. In this experiment the source of the ions are **potassium dihydrogen phosphate** (KH_2PO_4) and **sodium hydrogen phosphate** (Na_2HPO_4) respectively. Other salts which supply the same two ions in water solution may be used to form this buffer.

The two solid salts dissolve and form the required ions in water solution:



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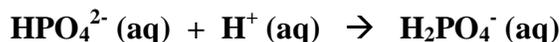
Phosphate Buffer (d1 Buffer) (Cont.)

The two active ions of the buffer are components of the phosphoric acid dissociation chain. The successive dissociations² of phosphoric acid link **phosphoric acid (H₃PO₄)**, **dihydrogen phosphate ion (H₂PO₄⁻)**, **hydrogen phosphate ion (HPO₄²⁻)**, and **phosphate ion (PO₄³⁻)**:

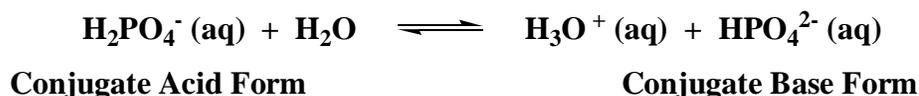


They two ions of the d1 buffer are related by being a **Brønsted** conjugate acid – conjugate base pair as shown in **Equation 2** above.

A buffer containing a conjugate acid – conjugate base pair is able to oppose pH change by reacting with either acid or base added to the solution. In the NBS d1 buffer, the reactions are:



These two ions, being a conjugate pair, are related by an acid dissociation equation with an associated acid dissociation (**K_a**) value; in this case, **K_a** is **K₂** of the phosphoric acid dissociation:



Writing the equilibrium constant expression for this reaction:

$$K_a = K_2 = \frac{[\text{H}_3\text{O}^+][\text{HPO}_4^{2-}]}{[\text{H}_2\text{PO}_4^-]}$$

Taking the negative of the logarithm to base 10 of both sides:

$$-\log_{10}(K_a) = -\log_{10}(K_2) = -\log_{10} \left(\frac{[\text{H}_3\text{O}^+][\text{HPO}_4^{2-}]}{[\text{H}_2\text{PO}_4^-]} \right)$$

Rearranging the equation:

$$-\log_{10}(K_a) = -\log_{10}(K_2) = -\log_{10} [\text{H}_3\text{O}^+] - \log_{10} \left(\frac{[\text{HPO}_4^{2-}]}{[\text{H}_2\text{PO}_4^-]} \right)$$

This Section Continues on the Next Page →

² Skoog and Others, Fundamentals of Analytical Chemistry, 8th Edition, Thomson.

Phosphate Buffer (d1 Buffer) (Cont.)

Using **p** notation ($\mathbf{p} = -\log_{10}$):

$$\mathbf{pK}_a = \mathbf{pK}_2 = \mathbf{pH} - \log_{10} \left(\frac{[\text{HPO}_4^{2-}]}{[\text{H}_2\text{PO}_4^-]} \right)$$

Rearranging the equation:

$$\mathbf{pH} = \mathbf{pK}_a (\mathbf{pK}_2) + \log_{10} \left(\frac{[\text{HPO}_4^{2-}]}{[\text{H}_2\text{PO}_4^-]} \right)$$

The value of \mathbf{K}_2 of phosphoric acid is 6.32×10^{-8} ; therefore $\mathbf{pK}_2 = 7.20$

The equation in this case is:

$$\mathbf{pH} = 7.20 + \log_{10} \left(\frac{[\text{HPO}_4^{2-}]}{[\text{H}_2\text{PO}_4^-]} \right)$$

This is a specific example of what is generally referred to as the **Henderson-Hasselbach Equation**, which may be quoted in a number of forms:

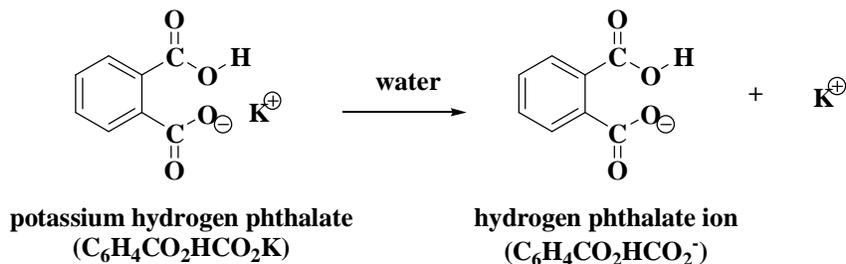
$$\mathbf{pH} = \mathbf{pK}_a + \log_{10} \left(\frac{[\text{salt}]}{[\text{acid}]} \right) \qquad \mathbf{pH} = \mathbf{pK}_a + \log_{10} \left(\frac{[\text{conjugate base}]}{[\text{conjugate acid}]} \right)$$

If the conjugate acid and the conjugate base form are present in **equal molarity** in the solution, the equation reduces to:

$$\mathbf{pH} = \mathbf{pK}_a \quad (\mathbf{pH} = \mathbf{pK}_2 = 7.20 \quad \text{in the case of the d1 buffer})$$

Phthalate Buffer (d2 Buffer)

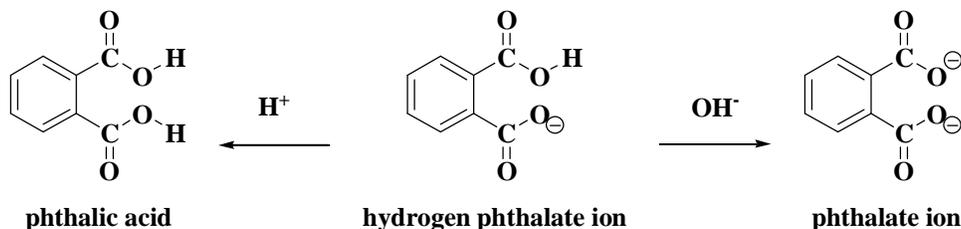
The NBS **d2** buffer solution contains **potassium hydrogen phthalate** ($\text{C}_6\text{H}_4\text{CO}_2\text{HCO}_2\text{K}$) as the buffer substance. This substance, also known as **KHP**, is used in analytical chemistry as a primary standard solid substance for acid-base titrations. It is available in very high purity and is convenient to store and use. In water, this substance forms ions:



This Section Continues on the Next Page →

Phthalate Buffer (d2 Buffer) (Cont.)

The hydrogen phthalate ion is able to react with either acid (H^+) or base (OH^-) as shown, producing respectively phthalic acid or phthalate ion. In this way, the solution is protected from pH change as long as the capacity of the buffer substance is not exceeded.



Gravimetric Titrations

Using a top-loading electronic balance, it is possible to do titrations quickly and more precisely than with a buret. This method is called **gravimetric titration**.

The procedure for a gravimetric titration is similar to that for a volumetric titration. But, instead of adding the titrating solution from a buret, the solution is added from a small plastic squeeze dropper bottle.

The squeeze dropper bottle has a narrow opening, allowing the titrating solution to be added one drop (or less) at a time. The amount of titrating solution dispensed is measured by a **mass change** rather than a volume change.

The titration calculations for a gravimetric titration require that the solution concentration in the dropper bottle be known on a mass basis; as **mmol titrant / g of solution** or as **mg titrant / g of solution** to give two possible examples.

The concentration of a gravimetric titration solution may be known from primary measurements of the mass of the reagent, if it is a primary standard substance, and the mass of the water solvent.

If the reagent is not a primary standard substance, the solution must be standardized by some other means, as in a volumetric titration.

In this Experiment, the titrating reagents are **0.1000 M NaOH** solution and **0.1000 M HCl** solution. Using data from the CRC Handbook, the concentration of these solutions in **mmol / g** units have been estimated as shown in the Table below. These are approximate values, but they are probably correct to within $\pm 0.2\%$ which is sufficient for our purpose.

Solution	mmol / mL	Density ³	mmol / g
0.1000 M NaOH	0.1000	1.0039 ⁴	0.0996
0.1000 M HCl	0.1000	1.0007 ⁵	0.0999

³ Approximate densities, Handbook of Chemistry and Physics, CRC Press, 54th Edition, 1973-1974.

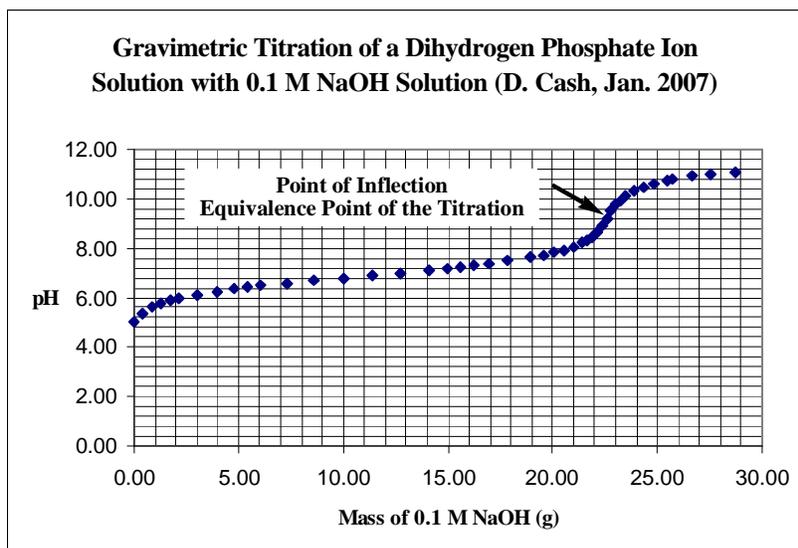
⁴ Density of 0.125 M NaOH solution at 20 °C.

⁵ Density of 0.137 M HCl solution at 20 °C.

Gravimetric Titration pH Curve for Dihydrogen Phosphate Ion

The graphic below shows a gravimetric titration curve obtained for a sample of a solution containing **dihydrogen phosphate ion** (H_2PO_4^-). The sample solution was titrated using a **pH meter**, by adding drops of **0.1000 M NaOH** solution from a squeeze dropper bottle.

The graphic shown plots the **pH** versus the **mass** of NaOH solution added. This is called a **gravimetric titration pH curve**.



From the start, the mass of the bottle and the pH of the solution were measured and recorded at every addition. The mass of titrant added at each point is calculated by **subtracting** the mass of the squeeze bottle at that point from the original mass of the bottle, as shown in the **Example 1** in the **Sample Calculations** on the next page.

Equivalence Point – Point of Inflection

The equivalence point of the pH titration, indicated by the arrow tip on the graphic, is the point of inflection of the curve of pH versus titrant amount. At the point of inflection, the **rate of change** of pH versus titrant amount goes from positive to negative.

The point of inflection may be estimated visually from the curve, or by graphical or mathematical analysis of the titration data. The simple visual method will be used in this Experiment. This is illustrated in the **Sample Calculations** that follow.

End-Point vs Equivalence Point of a Titration

The **end-point** of a titration is the experimentally determined end of the titration. The **equivalence point** is the theoretical point at which the reactants have been added in the exact stoichiometric ratio of the balanced equation of the reaction.

These terms are sometimes used as if they were the same, but in principle they are not. In a good analytical method, they will be very close to being the same point.

Sample Calculations

Example 1

In a gravimetric titration, the observed mass of the bottle containing the titrant solution **decreases** as the titration is carried out. This is illustrated in the first column (**Mass of Bottle**) of the Table of data below. This column and the **pH** column are the actual experimental measurements.

The mass of titrant solution that has been added to each point must be **calculated** by a **subtraction**. A portion of the mass and pH measurements observations for a gravimetric titration is given in the Table below. Calculate the value of the mass of titrant added at the final point in the Table.

In this experiment, the x-axis values of titrant mass are all calculated in this way for plotting.

Answer

Mass of Bottle (g)	Calculated Mass of Titrant Added (g)	pH
52.27	0.00	4.94
52.04	0.23	5.19
51.85	0.42	5.38
51.57	0.70	5.54
51.13	1.14	5.75
50.24	?	6.00

Example Data from Michael Deley, 1BT 2007:

The **mass** of the reagent bottle containing the titrating solution **decreases** as the titration proceeds. The **cumulative mass** of titrant solution **added** to each point must be **calculated** at each point by **subtracting** the measured mass at that point from the original mass.

Example: Calculated Mass of Titrant Added (final row in the Table)

$$= 52.27 \text{ g} - 50.24 \text{ g} = \underline{2.03} \text{ g}$$

The values for the x-axis of the plot are in the calculated column.

The Sample Calculations Section Continues on the Next Page →

Sample Calculations (Cont.)

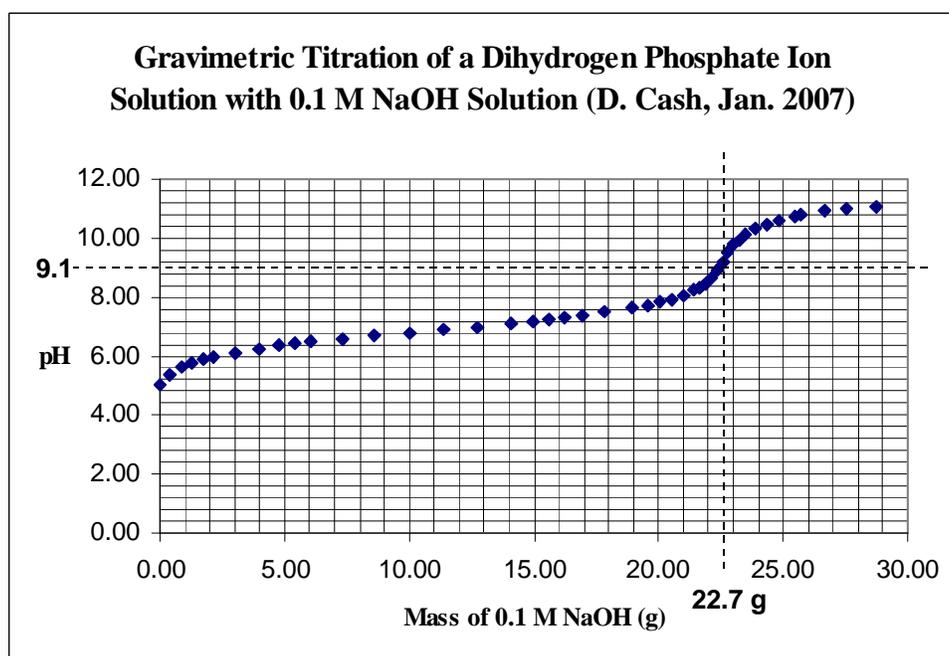
Example 2

The gravimetric titration curve on page 8, reproduced below, was obtained by titrating a **0.2786 g** sample of a pure crystalline solid containing the dihydrogen phosphate ion with **0.1000 M NaOH** solution.

Estimate the approximate **pH** to the nearest **0.1** unit and the approximate added titrant mass (**g**) to the nearest **0.1 g** that were added up to the point of inflection (equivalence point) of the gravimetric titration.

Answer

Estimate the **pH** and **Titrant Mass** approximately as shown by the dotted lines in the graphic below:



The equivalence point of the titration (estimated approximately) is at:

pH = 9.1

22.7 g of 0.1 M NaOH added.

The Sample Calculations Section Continues on the Next Page →

Sample Calculations (Cont.)

Example 3

The mass of titrant added to the equivalence point of the gravimetric titration on page 8 is **22.7 g**.

Calculate the amount of added **NaOH** in **mmol** units that has been added at the point of inflection (equivalence point). State the result to 3 significant figures.

Answer

Use the datum from the Table on page 7. The concentration of the **0.1 M NaOH** solution in mass terms is **0.0996 mmol / g**.

$$\text{mmol NaOH} = 22.7 \text{ g } 0.1 \text{ M NaOH} \times \frac{0.0996 \text{ mmol NaOH}}{1 \text{ g } 0.1 \text{ M NaOH}}$$

$$\text{mmol NaOH} = \underline{2.26} \text{ mmol NaOH}$$

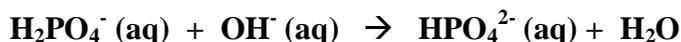
Example 4

From the amount of added **NaOH** in **mmol** units that has been added at the point of inflection (equivalence point) of the gravimetric titration (**2.26 mmol NaOH**), determine the amount of **dihydrogen phosphate ion (H₂PO₄⁻)** originally present in **mmol** units. State the result to 3 significant figures.

Answer

See the discussion of the phosphate buffer system on page 4.

It may be concluded that the net-ionic equation of the reaction of the titration is:



The stoichiometry of the reaction is **1 mol NaOH = 1 mol H₂PO₄⁻**.

$$\text{mmol H}_2\text{PO}_4^- = 2.26 \text{ mmol NaOH} \times \frac{1 \text{ mol H}_2\text{PO}_4^-}{1 \text{ mol NaOH}}$$

$$\text{mmol H}_2\text{PO}_4^- = \underline{2.26} \text{ mmol H}_2\text{PO}_4^-$$

The Sample Calculations Section Continues on the Next Page →

Sample Calculations (Cont.)

Example 5

The molar mass of **dihydrogen phosphate ion** (H_2PO_4^-) is **96.99 g / mol**.

Convert the amount **2.26 mmol H_2PO_4^-** present into **g** units.

State the result to 3 significant figures.

Answer

Convert **2.26 mmol H_2PO_4^-** into **mol** units and then into **g** units:

$$\begin{aligned} \text{g H}_2\text{PO}_4^- &= 2.26 \text{ mmol H}_2\text{PO}_4^- \times \frac{1 \text{ mol H}_2\text{PO}_4^-}{1000 \text{ mmol H}_2\text{PO}_4^-} \times \frac{96.99 \text{ g}}{1 \text{ mol H}_2\text{PO}_4^-} \\ &= \underline{0.219 \text{ g H}_2\text{PO}_4^-} \end{aligned}$$

Example 6

The **0.219 g dihydrogen phosphate ion** (H_2PO_4^-) was part of a **0.2786 g** sample of a pure crystalline solid (see **Example 1** on page 9).

Determine the percentage by mass (% w / w) of the **dihydrogen phosphate ion** in the substance. State the result to 3 significant figures.

Answer

$$\begin{aligned} \text{Percent by Mass Dihydrogen Phosphate Ion} &= \frac{\text{Mass H}_2\text{PO}_4^- \text{ (g)}}{\text{Mass Sample (g)}} \times 100 \% \\ &= \frac{0.219 \text{ g H}_2\text{PO}_4^-}{0.2786 \text{ g Sample}} \times 100 \% = \underline{78.7 \% \text{ w / w}} \end{aligned}$$

The Calculation Steps of Examples 3 to 6 May be Completed in a Single Step:

$$\begin{aligned} \text{Percent by Mass Dihydrogen Phosphate Ion} &= \underbrace{\text{mass 0.1 M NaOH (g)}}_{\text{Converts to mmol of NaOH}} \times \frac{0.0996 \text{ mmol NaOH}}{1 \text{ g 0.1 M NaOH}} \times \frac{1 \text{ mmol H}_2\text{PO}_4^-}{1 \text{ mmol NaOH}} \\ &\times \underbrace{\frac{0.09699 \text{ g H}_2\text{PO}_4^-}{1 \text{ mmol H}_2\text{PO}_4^-}}_{\text{Converts to g of H}_2\text{PO}_4^-} \times \underbrace{\frac{100 \%}{\text{Sample Mass (g)}}}_{\text{Converts to \% w / w of H}_2\text{PO}_4^-} \end{aligned}$$

Name		Day		Start Time	
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PRE-LABORATORY PREPARATION

To be completed before the laboratory session.

To be submitted before beginning the experiment (15 points).

Plotting Exercise (5 points)

Use software (e.g. Microsoft Excel) to plot the following set of data points with **Temperature** on the x-axis and **pH** on the y-axis.

0.0100 M Sodium Borate (Borax) Buffer		0.0100 M Sodium Borate (Borax) Buffer	
Temperature (°C)	pH	Temperature (°C)	pH
10	9.332	25	9.180
15	9.276	30	9.139
20	9.225	35	9.102

Instructions See the similar plots on page 18.

- Select Chart Type: **X-Y Scatter Plot with No Lines**.
- The pH scale must be such that the pH can be estimated to the third decimal place from any given temperature value. You can force Excel to display three decimal places by selecting the scale, and formatting the values as numbers.
- Add a trendline to your plot: choose a **polynomial of order 2** for the data set.
- Customize your plot by adding: a title including **your name and the date**; scale labels; major and minor grid lines; tick marks.
- Print your plot and bring a copy with you to the laboratory; you will need it during the laboratory period.

Show your plot to the instructor for initialing before starting the laboratory.

Keep the plot for use during the laboratory and for preparing your report.

The PRE-LABORATORY PREPARATION Continues on the Next Page →

PRE-LABORATORY PREPARATION (Cont.)

Questions: Answer in the space provided. **Show work.**

Q-1 Fill in the **three** missing values in the Table using your Mohawk ID **nnnnnnXYZ** (1 point).

See **Example 1** on page 9.

Mass of Bottle (g)	Mass of Bottle (g)	Calculated Mass of Titrant Added (g)	pH
47.3Y		0.00	4.94
47.0Z			5.19

Q-2. Calculate the quantity (g) of **sodium hydrogen phosphate** (Na_2HPO_4), molar mass **141.96 g / mol**, required to prepare **500 mL (0.5000 L)** of a **0.0249 M** solution. State the value to two places after the decimal point. Enter the mass value in **Table B** in the **DATA TABLES AND REPORT** section. (3 points) Show work.

Required Mass of Sodium Hydrogen Phosphate (g) = _____ g

Q-3. Calculate the quantity (g) of **potassium dihydrogen phosphate** (KH_2PO_4), molar mass **136.09 g / mol**, required to prepare **500 mL (0.5000 L)** of a **0.0249 M** solution. State the value to two places after the decimal point. Enter the mass value in **Table B** in the **DATA TABLES AND REPORT** section. (3 points) Show work.

Required Mass of Potassium Dihydrogen Phosphate (g) = _____ g

Q-4. Calculate the quantity (g) of **potassium hydrogen phthalate** ($\text{C}_6\text{H}_4\text{CO}_2\text{HCO}_2\text{K}$), molar mass **204.23 g / mol**, required to prepare **250 mL (0.2500 L)** of a **0.0496 M** solution. State the value to two places after the decimal point. Enter the mass value in **Table B** in the **DATA TABLES AND REPORT** section. (3 points) Show work.

Required Mass of Potassium Hydrogen Phthalate (g) = _____ g

PRE-LABORATORY PREPARATION

Total = / 15

PROCEDURE

In this Experiment you will work with a partner or in a small group, as instructed. Record the name(s) of your partner(s) in **Table B** in the **DATA TABLES AND REPORT** section.

A. Preparation of Glassware and Apparatus

The following **clean** glassware and laboratory apparatus is required for this part of the experiment:

- | | | |
|---|--|--|
| For each pair or group: | <input type="checkbox"/> two long-stem funnels | <input type="checkbox"/> a 250 mL and a 500 mL volumetric flask and their stoppers |
| <input type="checkbox"/> two Teflon coated magnetic stirring bars | <input type="checkbox"/> two small beakers | <input type="checkbox"/> two 250 mL beakers |
| <input type="checkbox"/> two glass stirring rods | <input type="checkbox"/> a spatula | <input type="checkbox"/> a digital thermometer (extra apparatus, shared) |
| | <input type="checkbox"/> a dropper pipet | |

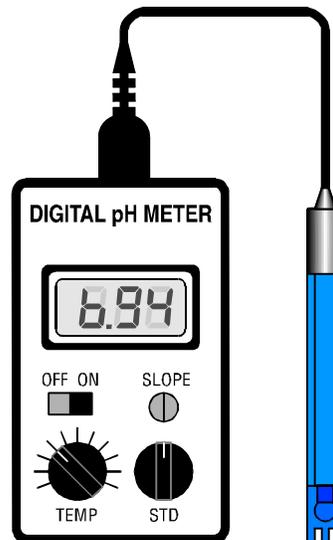
- A-1. Clean the glassware and apparatus if necessary with a 1 % solution of detergent in warm water. See **Cleaning and Drying of Glassware** on page **Error! Bookmark not defined.**. Rinse the cleaned glassware and apparatus with tap water and then with distilled water. To avoid breakage, do not leave any glassware standing in an unstable position.

pH Meters

Most of the pH meters used by the department are microprocessor-controlled digital meters with the calibration points for several standard buffer solutions built into memory. The electrodes are usually the combination type.

Some of the pH meters can accommodate a temperature probe for reading and correcting for temperature differences. Otherwise, the solution temperature can be set manually.

Note: The pH meter program corrects for changes in actual pH buffer values with temperature and for changes in the Nernst slope.



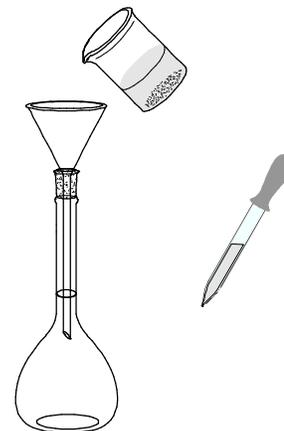
Older Manual-Set pH Meter

B. Preparation of NBS Standard Buffer Solutions

Verify that the names, the formulas, and the molar masses of the salts provided (listed on the bottle labels) match those in the instructions. If other salts or hydrates are substituted, the instructor will provide the required masses for solution preparation.

- B-1. Use two clean, dry weighing bottles or weighing boats. **Do trial weighings using a top-loading balance, and final weighings using a four-decimal analytical balance.**

Weigh by addition, the amounts of the **sodium hydrogen phosphate** and the **potassium dihydrogen phosphate** required to prepare the **Phosphate d1 Buffer** solution. Each of the two masses taken must be within ± 0.01 g of the required mass. Record the measured mass values to 4 places after the decimal point in **Table B** on page 29 in the **DATA TABLES AND REPORT** section.



- B-2. Use a wash bottle, a small beaker, and a funnel to completely transfer both of the reagents into a single clean **500 mL** volumetric flask.

Fully dissolve the solids and make the volume up to the fill mark with distilled water from a dropper pipet. Ensure that the solution is completely uniform. Label the flask **Phosphate d1 Buffer**. Cap the flask while not in use.

- B-3. Use a clean, dry weighing bottle or a clean, dry weighing boat. **Do trial weighings using a top-loading balance, and final weighings using a four-decimal analytical balance.**

Weigh by addition, the amount of the **potassium hydrogen phthalate** required to prepare the **Phthalate d2 Buffer** solution. The mass taken must be within ± 0.01 g of the required mass. Record the measured mass values to 4 places after the decimal point in **Table B**.

- B-4. Use a wash bottle, a small beaker, and a funnel to completely transfer the reagent into a clean **250 mL** volumetric flask.

Fully dissolve the solid and make the volume up to the fill mark with distilled water from a dropper pipet. Ensure that the solution is completely uniform. Label the flask **Phthalate d2 Buffer**. Cap the flask while not in use.

- B-5. Use several small portions of the **Phosphate d1 Buffer** solution to rinse a clean **250 mL** beaker and a Teflon-coated magnetic stirring bar. Collect the rinse in a waste beaker for disposal later. Place about **100 mL** of the buffer solution in the beaker. Label the beaker **Phosphate d1 Buffer**.

- B-6. Repeat with the **Phthalate d2 Buffer** solution in a second **250 mL** beaker with a second Teflon-coated magnetic stir bar. Label the beaker **Phthalate d2 Buffer**.

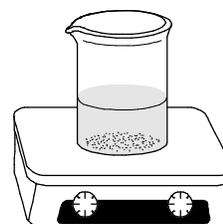
C. Calibration of a pH Meter

- C-1. Both of the NBS standard buffer solutions have pH values that are temperature dependent. See page 18. A digital thermometer will be supplied. Use the thermometer to measure the laboratory air temperature and the temperature of the two buffer solutions to the nearest 0.1 °C. **Do not cross-contaminate the buffer solutions.** Record the temperature values in **Table C** in the **DATA TABLES AND REPORT** section.

The air and solution temperatures will probably be similar, or identical. If they are not, consult the instructor as to whether to use the laboratory air temperature or a mean temperature for the next steps.

Note: The pH meter can be interfaced with a temperature sensing probe. However, the probe is both delicate and expensive, so it will not be used for this experiment.

- C-2. The pH meter assigned to you may already have a stirring motor and a clamp stand. If not, obtain a stirring motor, a retort stand and an extension clamp. Set up the apparatus for use with the supplied pH meter. The clamp will be used to hold the electrodes in the solution but **safely above** the stirring bar. The instructor will demonstrate.



Stirring Motor

The delicate tip of the electrode must **NEVER** be scratched or allowed to contact any object.

DO NOT touch the delicate tip of the electrode with anything other than distilled water or a test solution in a beaker. Combination pH electrodes must **NEVER** be allowed to remain out of an aqueous solution for more than a few minutes. They will always be in use in a test solution, or left in distilled water. If they become dry it may be difficult to put them back into use.

- C-3. Raise the combination electrodes of the pH meter from its distilled water beaker. Rinse the electrodes with a stream of distilled water from a wash bottle; collect the rinse in the waste beaker. Allow the water to drip off the electrodes for a minute.
- C-5. Carefully place the electrodes in the **Phosphate d1 Buffer** solution.
- C-6. Stir the buffer solution gently by using the magnetic stirrer. **MAKE SURE** that the stirring bar does not hit and damage the electrodes.
- C-7. If necessary, plug the pH meter in to a **120 V AC** outlet. Turn the meter **ON**. Ensure that the meter is set to the **pH** mode, and not to the **mV** mode.
- C-8. Set the temperature control on the pH meter to the laboratory temperature determined in **Step C-1** above.

This Section Continues on the Next Text Page →

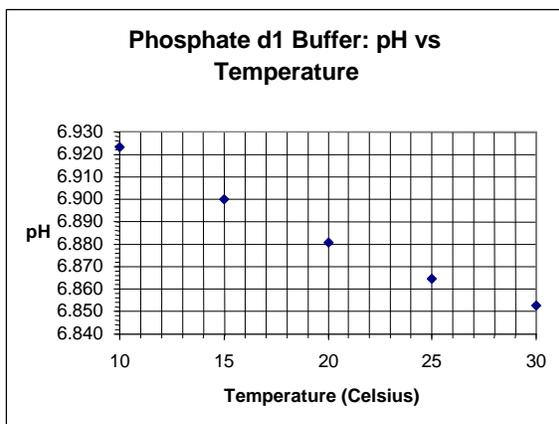
NBS Standard Buffers

The Table below gives the literature values for the pH of the **NBS Standard Phosphate d1 Buffer** and the **NBS Standard Phthalate d2 Buffer** as a function of temperature. This data is in the memory of a microprocessor controlled pH meter, and is utilized in the calibration process. The plots below are respectively: Excel X-Y scatter plots with no connecting lines of the two sets of data points, and X-Y plots with a polynomial trendline of order 2.

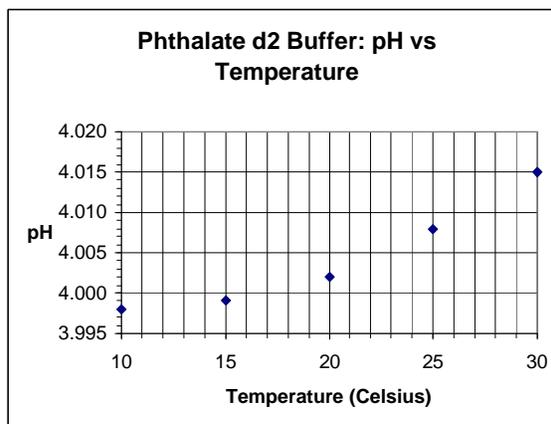
Phosphate d1 Buffer	
Temperature (°C)	pH
10	6.923
15	6.900
20	6.881
25	6.865
30	6.853

Phthalate d2 Buffer	
Temperature (°C)	pH
10	3.998
15	3.999
20	4.002
25	4.008
30	4.015

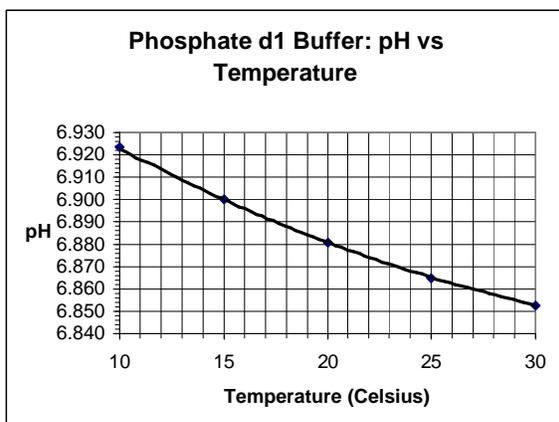
**Phosphate d1 Buffer: X-Y Scatter Plot
No Connecting Lines**



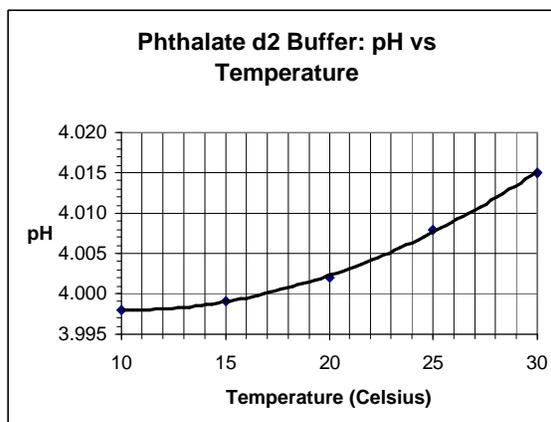
**Phthalate d2 Buffer: X-Y Scatter Plot
No Connecting Lines**



**Phosphate d1 Buffer: X-Y Scatter Plot
Trendline: Polynomial of Order 2**



**Phthalate d2 Buffer: X-Y Scatter Plot
Trendline: Polynomial of Order 2**



C. Calibration of a pH Meter (Cont.)

These instructions are specific to the **Hanna Instruments HI8147** pH meter. If you are using a different instrument, specific instructions will be supplied.

- C-9. ? Press the **CAL** key. The **pH** symbol will flash or blink, indicating that the instrument is in Calibration mode. The **pH** symbol will be flashing during the entire calibration.

The meter should sense contact with a solution of pH appropriate for the **Phosphate d1 Buffer** solution, when the **d1** symbol should appear in the display window. The symbol **d1** is the designation for the NBS standard **Phosphate d1 Buffer** solution.

? Press the **CON** key (**Confirm**) to confirm that this is the **Phosphate d1 Buffer** solution. The correct pH value will be displayed **with the temperature correction made**.

Wait for a minute.

? Press the **CON** key again to accept this value into memory.

Note: There is an alternate set of standard buffers, the “**t**” set, which may be used to calibrate the meter. There is a procedure for instructing the meter to use a **t** buffer pH setting at this stage if necessary.



**Microprocessor-Controlled
Digital pH Meter
(Hanna Instruments HI8417)**

Photo: Emily Girard 3CH 2005

This Section Continues on the Next Page →

C. Calibration of a pH Meter (Cont.)

- C-10. Raise and rinse the electrodes with a stream of distilled water from a wash bottle; collect the rinse in the waste beaker. Allow the water to drip off the electrodes for a minute. Carefully place the electrodes in the **Phthalate d2 Buffer** solution. Stir gently.

The meter should sense contact with a solution of pH appropriate for the **Phthalate d2 Buffer** solution, when the **d2** symbol should appear in the display window. The symbol **d2** is the designation for the **NBS standard Phthalate d2 Buffer** solution.

? Press the **CON** key (**Confirm**) to confirm that this is the **Phthalate d2 Buffer** solution. The correct pH value will be displayed **with the temperature correction made**.

Wait for a minute.

? Press the **CON** key again to accept this value into memory.

- C-11. ? Press the **CON** key again to exit the calibration mode.
The **pH** symbol should now stop flashing. The meter has been calibrated.
- C-12. Raise and rinse the electrodes with a stream of distilled water from a wash bottle, and place the electrodes in distilled water during wait times.
- C-13. Use the calibrated pH meter to measure the pH of the following:
a. a fresh sample of the **Phosphate d1 Buffer**;
b. a fresh sample of the **Phthalate d2 Buffer**;
c. two samples of the **0.0100 M Sodium Borate (Borax) Buffer** solution provided.
Record the pH values in **Table C** on page 30.
- C-14. Determine the literature values of the pH of the two NBS Standard buffer solutions at the laboratory temperature to 3 places after the decimal point using the plots in **Table C** on page 31. Record the literature values in **Table C** on page 30.
Show on the plots how the literature value was determined.
- C-15. Determine the literature value of the pH of the borax buffer solution at the laboratory temperature to 3 places after the decimal point using your **Pre-Laboratory Preparation** plot. Record the literature value in **Table C** on page 30.
Show on the plot how the literature value was determined.
Attach the plot to your report.
- C-16. If there is a major discrepancy between the literature pH values and the experimental pH values, consult the instructor.

**D. Preparing for a Gravimetric pH Titration
– Dihydrogen Phosphate Ion and 0.1000 M NaOH**

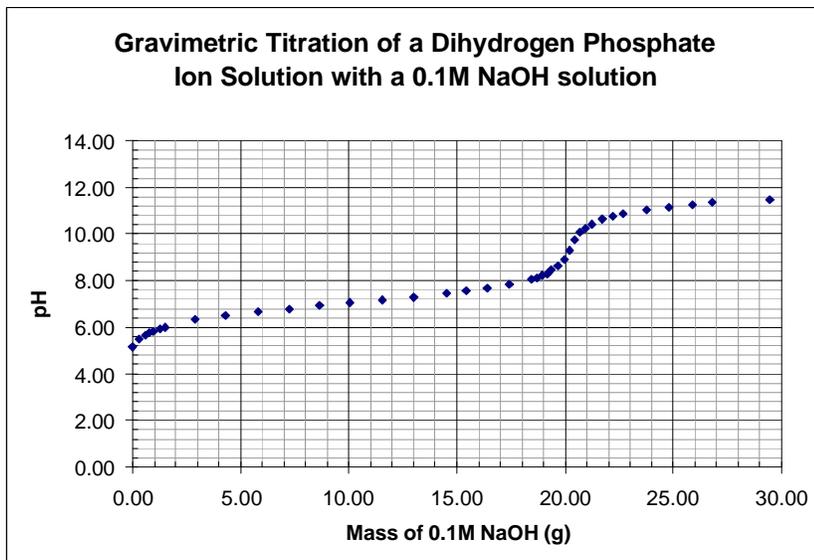
D-1. You will need the following apparatus and clean labware for the pH titration:

- | | | |
|--|--|---|
| <input type="checkbox"/> a Teflon-coated magnetic stirring bar | <input type="checkbox"/> a small beaker | <input type="checkbox"/> a stirring motor |
| <input type="checkbox"/> a 250 mL beaker | <input type="checkbox"/> a combination electrode | <input type="checkbox"/> a clamping stand for the electrode |
| <input type="checkbox"/> a pH meter | | |

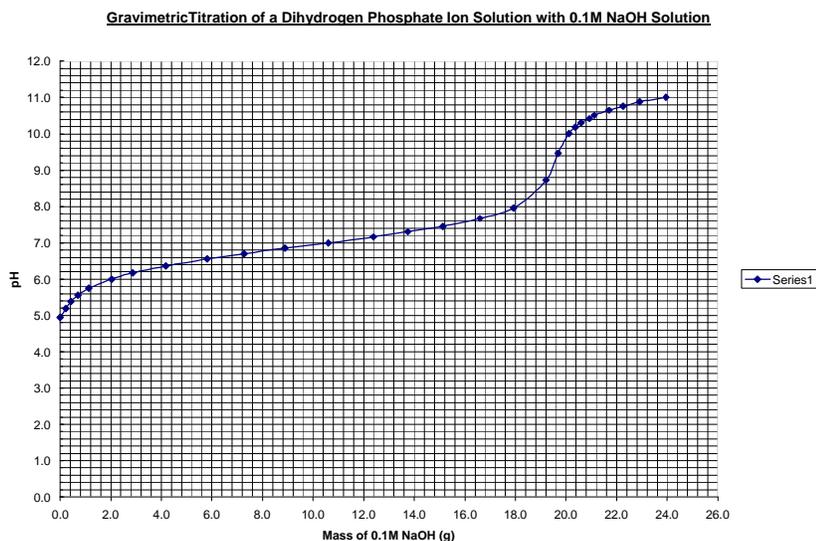
Each pH meter position should have available a small polymer squeeze-dropper bottle labeled as **0.1000 M NaOH** solution.

- D-2. Weigh out accurately **by analytical balance** between **0.22 – 0.24 g** of the crystalline solid labelled **A**. The solid **A** contains dihydrogen phosphate ion. Record the mass values **to four places after the decimal point** on page 33 in **Table E** in the **DATA TABLES AND REPORT** section.
- D-3. Transfer the solid **A** **quantitatively** into a clean **250 mL** beaker labelled **A**. Add distilled water to the **100 mL** mark of the beaker. **Solution A** is now ready to be titrated.
- D-4. If necessary, refill the small polymer squeeze-dropper bottle from the stock bottle of standard **0.1000 M** sodium hydroxide solution. Cap the bottle and dry the outside if necessary.
- D-5. Place the **250 mL** beaker containing **Solution A** onto the stirring motor. Add a clean Teflon-coated magnetic stirring bar to the beaker. Stir gently.
- D-6. Raise the combination electrodes of the pH meter from the distilled water beaker. Rinse the electrodes with distilled water from a wash bottle; collect the rinse in the waste beaker. Place the electrodes carefully into the beaker, and clamp it in a safe position above the stirring bar.
- D-7. Turn on the pH meter. The initial pH of the titration will be about **5**, as illustrated in the student titration curves on the next pages.

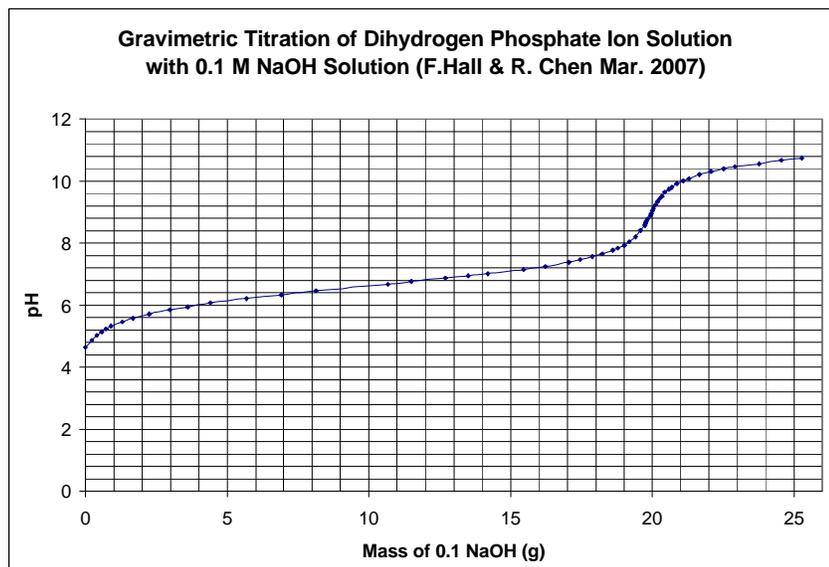
**Data Collected
And Plotted by
Abitab Rangunathan
(1BT 2007)**



**Data Collected
And Plotted by
Paula Gremmen
(1BT 2007)**



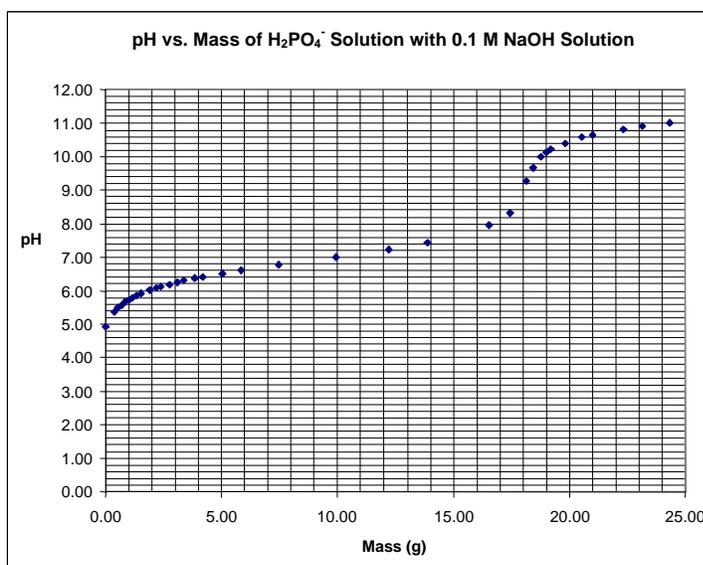
**Data Collected
And Plotted by
Francois Hall
And Richard Chen
(1BT 2007)**



E. Gravimetric pH Titration of Dihydrogen Phosphate Ion Solution with 0.1000 M NaOH Solution

- E-1. Dry the outside of the small squeeze dropper bottle containing **0.1000 M** NaOH. Zero a **top-loading** balance and weigh the bottle plus its content of NaOH solution. Record the measured starting mass value to **2 places after the decimal point** on page 32 in **Table E** in the **DATA TABLES AND REPORT** section.
- E-2. The stirring bar should be rotating slowly to mix the solution continuously. Read the starting pH value measurement to **2 places after the decimal point** for the solution and record it on page 32 in **Table E**.
- E-3. Add **5 drops** of the NaOH solution from the **0.1000 M NaOH** squeeze dropper bottle to the solution in the beaker. Wait for the pH reading to stabilize, which should not take more than about **15** seconds at most. Record the new pH value measurement for the solution in **Table E**.
- E-4. Zero a top-loading balance and weigh the bottle plus contents again. Record the new measured mass value to 2 places after the decimal point in **Table E**.
- E-5. Continue to titrate by counting the addition of drops of the solution from the squeeze dropper bottle. Measure and record the **pH** and the **mass** of the bottle after each addition.
- At the beginning, **and near the end-point**, when the pH is changing rapidly, make small additions of **5** or **10** drops at a time. When the pH of the solution is not changing rapidly, make large additions, up to **25** or **30** drops at a time. Use the curves on the previous page and the one below as a guide.
- E-6. Continue the titration until the pH of the solution remains fairly constant at or above **pH = 11**.

**Data Collected
And Plotted by
Khaalid Ali
(1BT 2007)**



F. Preparing for a Gravimetric pH Titration – Carbonate Ion and 0.1000 M HCl

F-1. You will need the following apparatus and clean labware for the pH titration:

- a Teflon-coated magnetic stirring bar
- a small beaker
- a stirring motor
- a **250 mL** beaker
- a combination electrode
- a clamping stand for the electrode
- a pH meter

Each pH meter position should have available a small polymer squeeze-dropper bottle labeled as **0.1000 M HCl** solution.

F-2. Weigh out accurately by **analytical balance** between **0.11 – 0.12 g** of the crystalline solid labelled **B**. Record the mass values **to four places after the decimal point** on page 35 in **Table G** in the **DATA TABLES AND REPORT** section.

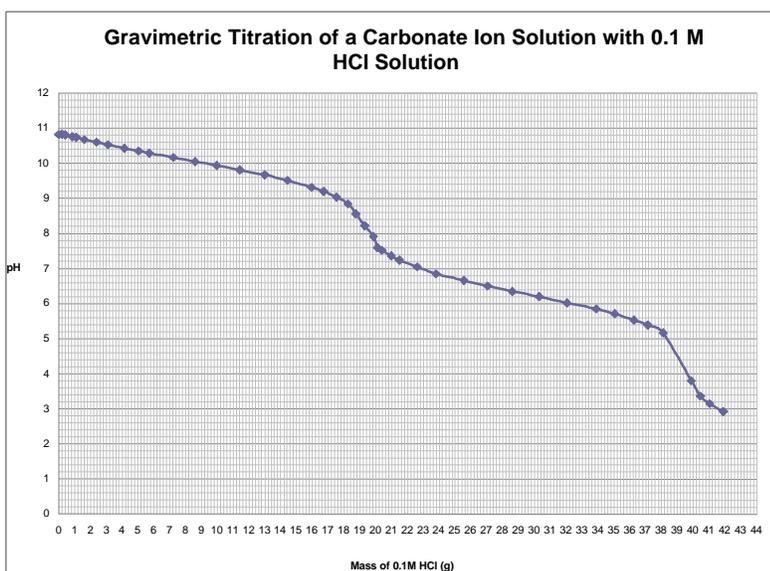
F-3. The solid **B** contains carbonate ion. Transfer the solid **quantitatively** into a clean **250 mL** beaker labelled **B**. Add distilled water to the **100 mL** mark of the beaker. **Solution B** is now ready to be titrated.

F-4. If necessary, refill the small polymer squeeze-dropper bottle from the stock bottle of standard **0.1000 M** hydrochloric acid solution. Cap the bottle and dry the outside if necessary.

F-5. Place the **250 mL** beaker containing **Solution B** onto the stirring motor. Add a clean Teflon-coated magnetic stirring bar to the beaker. Stir gently.

F-6. Raise the combination electrodes of the pH meter from the distilled water beaker. Rinse the electrodes with distilled water from a wash bottle; collect the rinse in the waste beaker. Place the electrodes carefully into the beaker, and clamp it in a safe position above the stirring bar.

**Data Collected
And Plotted by
Sara Pereira
(1BT 2007)**



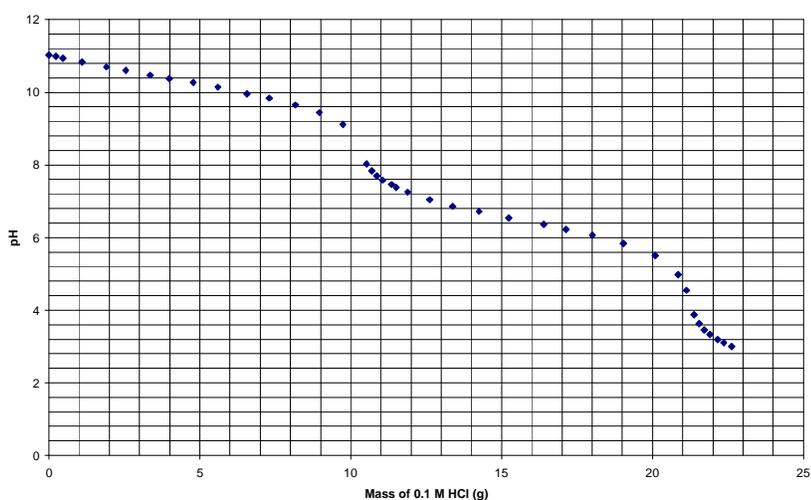
G. Gravimetric pH Titration of Carbonate Ion Solution with 0.1000 M HCl Solution

- G-1. Dry the outside of the small squeeze dropper bottle containing **0.1000 M HCl**. Zero a **top-loading** balance and weigh the bottle plus its content of HCl solution. Record the measured starting mass value to **2 places after the decimal** on page 34 in **Table G** in the **DATA TABLES AND REPORT** section.
- G-2. The stirring bar should be rotating slowly to mix the solution continuously. Read the starting pH value measurement to **2 places after the decimal** for the solution and record it on page 34 in **Table G**.
- G-3. Add **5 drops** of the HCl solution from the squeeze dropper bottle to the solution in the beaker. Wait for the pH reading to stabilize, which should not take more than about **15 seconds** at most. Record the new pH value measurement for the solution in **Table G**.
- G-4. Zero a top-loading balance and weigh the bottle plus contents again. Record the new measured mass value in **Table G**.
- G-5. Continue to titrate by counting the addition of drops of the solution from the squeeze dropper bottle. Measure and record the pH and the mass of the bottle after each addition.

At the beginning, **and near the end-points**, when the pH is changing rapidly, make small additions of **5** or **10** drops at a time. When the pH of the solution is not changing rapidly, make large additions, up to **25** or **30** drops at a time. Use the curve on page 24, the one below and the Figure in **Harris (Chapter 11, page 233, Figure 11-3)** as a guide.

- G-6. Continue the titration until the pH of the solution remains fairly constant at or below **pH = 3**.

Gravimetric Titration of Carbonate Ion Solution With 0.1 M HCl Solution



Data Collected
And Plotted by
Vincent Waite
(1BT 2007)

REPORT

The titration data and the plots may be submitted jointly by the group. If submitting individually, a photocopy of the titration data is sufficient. All other parts of the report are to be submitted individually.

Table B

- R-1. Determine and enter the **experimental mass (g)** of each of the solids used to prepare the buffer solutions in **Table B**.
- R-2. Calculate the **experimental molarity (mol / L)** of each of the buffer solution components. Enter the values in **Table B**. Show your calculation method for one example.
- R-3. Calculate the **percentage error** compared to the nominal molarity for each component. Enter the values in **Table B**.

Table C

- R-4. Estimate the **literature pH** for the two NBS Standard buffers to 3 places after the decimal point using the two given plots of pH versus temperature, at the mean temperature as determined during the experiment. Enter the two values in **Table C**.
- R-5. Estimate the **literature pH** for the **0.0100 M Borax** buffer to 3 places after the decimal point using your Pre-Laboratory Preparation plot of pH versus temperature, at the mean temperature as determined during the experiment. Enter the value in **Table C**.
- R-6. Determine the absolute error (difference) between the literature pH and the experimental pH of each of the three buffer solutions when measured after the calibration. Enter the values in **Table C**.

The REPORT Section Continues on the Next Page →

REPORT (Cont.)

Table E

- R-7. Determine and enter the mass of **Substance A** (g).
- R-8. Transcribe your experimental observations of bottle mass and pH into a spreadsheet Table (e.g. Microsoft Excel) with the format shown:

Mass of Bottle (g)	Calculated Mass of Titrant Added (g)	pH
Etc.	Etc.	Etc.

Use a calculation formula in the cells to determine the **Calculated Mass of Titrant Added** for each observation point. See **Example 1** on page 9.

- R-9. Use the software (e.g. Microsoft Excel) to plot the data for your gravimetric titration with **Calculated Mass of 0.1 M NaOH Titrant Added (g)** on the x-axis and **pH** on the y-axis.
- The pH scale must be such that the pH can be estimated to the first decimal place from any given mass value (Excel will do this automatically).
 - The mass scale must be such that the mass can be estimated to the first decimal place from any given pH value (Excel will do this automatically).
 - Choose an **X – Y scatter plot** for the plot. **DO NOT** add a trend line to the plot. Either choose to have **no lines** joining the points, or choose to have **a smooth line** joining the points.
 - Customize** the plot by adding: major and minor grid lines; major and minor tick lines; a title, including **your name(s) and the date**; scale labels.
 - Print the data table and the plot. If possible, place the data table and the plot on a single page. The data table and plot are to be attached to the report.**
- R-10. The point of inflection of the plot is the end-point and also the equivalence point of the gravimetric titration. Estimate the experimental **pH** to the nearest **0.1** unit and the experimental added titrant mass (**g**) to the nearest **0.1 g** that were added up to the point of inflection. See **Example 2** on page 10.
- R-11. Calculate the experimental percentage by mass of the **hydrogen phosphate ion (% w / w)** in the test solution. You may use the single step calculation as shown on page 12 and in **Table E** or follow the steps in **Examples 3 to 6** on pages 11 to 12.

The REPORT Section Continues on the Next Page →

REPORT (Cont.)

Table G

- R-12. Determine and enter the mass of **Substance B** (g).
- R-13. Transcribe your experimental observations of **Mass of Bottle** (g) and **pH** into a spreadsheet Table (e.g. Microsoft Excel) with the format shown:

Mass of Bottle (g)	Calculated Mass of Titrant Added (g)	pH
Etc.	Etc.	Etc.

Use a calculation formula in the cells to determine the **Calculated Mass of Titrant Added** for each observation point. See **Example 1** on page 9.

- R-14. Use software (e.g. Microsoft Excel) to plot the data for your gravimetric titration with **Calculated Mass of 0.1 M HCl Titrant Added** (g) on the x-axis and **pH** on the y-axis.
- The pH scale must be such that the pH can be estimated to the first decimal place from any given mass value (Excel will do this automatically).
 - The mass scale must be such that the mass can be estimated to the first decimal place from any given pH value (Excel will do this automatically).
 - Choose an **X – Y scatter plot** for the plot. **DO NOT** add a trend line to the plot. Either choose to have **no lines** joining the points, or choose to have **a smooth line** joining the points.
 - Customize** the plot by adding: major and minor grid lines; major and minor tick lines; a title, including **your name(s) and the date**; scale labels..
 - Print the data table and the plot. If possible, place the data table and the plot on a single page. The data table and plot are to be attached to the report.**
- R-15. The **second point of inflection** of the plot is the end-point and also the equivalence point of the gravimetric titration. Estimate the experimental **pH** to the nearest **0.1** unit and the experimental added titrant mass (g) to the nearest **0.1 g** that were added up to the second point of inflection. See **Example 2** on page 10 and the graphic on page 35.

Bonus Questions

See page 36 for the bonus point questions.

Name		Day		Start Time	
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DATA TABLES AND REPORT

The following **Tables** are to be used for recording observations and measurements. Measured values are to be recorded in the heavily shaded cells **IN INK**. Leave this page open on your bench during the experiment period. Have it initialed by the instructor on completing each section.

The completed and initialed **DATA TABLES AND REPORT** section must be handed in along with any additional pages you may submit as your report.

Table B: Preparation of NBS Standard Buffer Solutions

Name(s) of Partner(s): _____ + _____

Salts for Buffer Solution Preparation (Required Mass to 2 places after the decimal point)

Salt Name	sodium hydrogen phosphate	potassium dihydrogen phosphate	potassium hydrogen phthalate
Salt Formula	Na_2HPO_4	KH_2PO_4	$\text{C}_6\text{H}_4\text{CO}_2\text{HCO}_2\text{K}$
Molar Mass (g / mol)	141.96	136.09	204.23
Required Mass* (g)			

* The Required Mass values were calculated in the Pre-Laboratory Preparation.

Weighings of the Solid Salts (In Ink to 4 places after the decimal point)

Salt / Required* Mass (g)	Container Empty (g)	Container + Salt (g)	Experimental Mass of Salt (g)
sodium hydrogen phosphate _____ g			
potassium dihydrogen phosphate _____ g			
potassium hydrogen phthalate _____ g			

Instructor's Initials: _____ (5 points)

The DATA TABLES AND REPORT Section Continues on the Next Page →

DATA TABLES AND REPORT (Cont.)

Table B: Preparation of NBS Standard Buffer Solutions (Cont.)

Buffer Solution Concentrations (to at least 4 places after the decimal point)

Salt Name	sodium hydrogen phosphate	potassium dihydrogen phosphate	potassium hydrogen phthalate
Salt Formula	Na ₂ HPO ₄	KH ₂ PO ₄	C ₆ H ₄ CO ₂ HCO ₂ K
Molar Mass (g / mol)	141.96	136.09	204.23
Nominal Molarity (mol / L)	0.0249 M	0.0249 M	0.0496 M
Experimental Molarity (mol / L)			
Percentage Error (%)			

Sample Calculation: Show the Calculation of One of the Salt Concentrations. (3 points)

Concentration of _____ (mol / L) = _____ mol / L

Table C: Calibration of a pH Meter

Room Air Temperature = _____ °C d1 Solution Temperature = _____ °C

d2 Solution Temperature = _____ °C Mean Solution Temperature = _____ °C

Buffer Solution Data

Buffer Solution	Phosphate d1	Phthalate d2	0.0100 M Borate	0.0100 M Borate
Mean Solution Temperature (°C)	_____ °C	_____ °C	_____ °C	_____ °C
Literature pH At Solution Temperature				
Observed pH After Calibration of Meter				
Absolute Error in pH Value (? pH)				

Instructor's Initials on Completion: _____ (5 points)

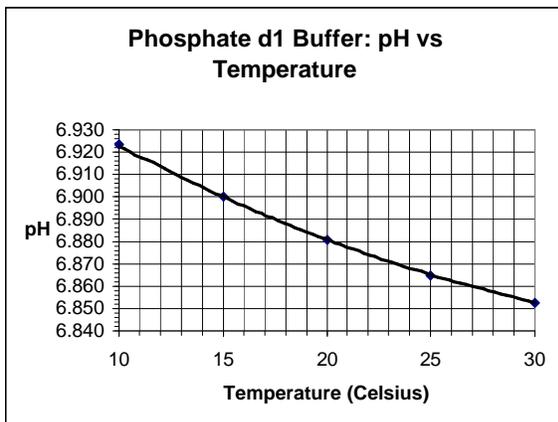
The DATA TABLES AND REPORT Section Continues on the Next Page →

Name		Day		Start Time	
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DATA TABLES AND REPORT (Cont.)

Table C: Calibration of a pH Meter (Cont.)

**Phosphate d1 Buffer: X-Y Scatter Plot
Trendline: Polynomial of Order 2**



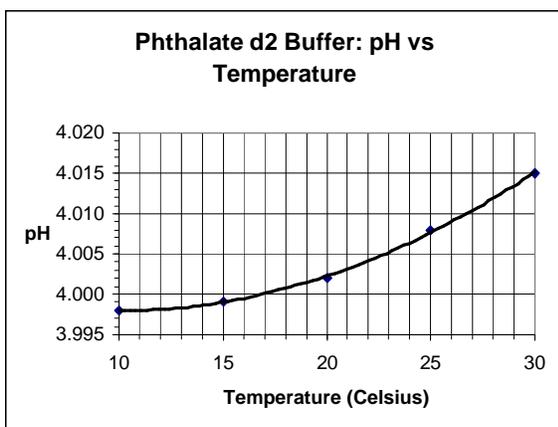
Estimation of the literature pH of the d1 buffer at the laboratory temperature.

Show on the plot how the pH was determined.

Solution Temperature (°C) _____ °C

Literature pH _____

**Phthalate d2 Buffer: X-Y Scatter Plot
Trendline: Polynomial of Order 2**



Estimation of the literature pH of the d2 buffer at the laboratory temperature.

Show on the plot how the pH was determined.

Solution Temperature (°C) _____ °C

Literature pH _____

The DATA TABLES AND REPORT Section Continues on the Next Page →

Name		Day		Start Time	
------	--	-----	--	------------	--

DATA TABLES AND REPORT (Cont.)

Table E: Gravimetric pH Titration of H₂PO₄⁻ Solution with 0.1000 M NaOH Solution (Cont.)

Weighings of Substance A (In INK to Four Places After the Decimal)

Initial Mass (g)	Final Mass (g)	Sample Mass (g)

Data and Plotting: See the **Report Instruction R-8** on page 27. See **Example 2** on page 10.

Estimated Mass of 0.1000 M NaOH Solution Added at the Inflection Point (g) = _____ g
(To nearest 0.1 g)

Estimated pH at the Equivalence Point = _____ pH (To nearest 0.1 unit)

Attach Your Data Table and Plot, showing how the values were obtained.

Calculate the experimental percentage by mass (% w / w) of the dihydrogen phosphate ion in the substance A. State the value to three significant figures. Show work. (7 points)

See **Examples 3 to 6** on pages 11 to 12 or use this one-step calculation:

$$\begin{aligned}
 \text{Percent by Mass Dihydrogen Phosphate Ion} &= \text{mass 0.1 M NaOH (g)} \times \underbrace{\frac{0.0996 \text{ mmol NaOH}}{1 \text{ g 0.1 M NaOH}}}_{\text{Converts to mmol of NaOH}} \times \underbrace{\frac{1 \text{ mmol H}_2\text{PO}_4^-}{1 \text{ mmol NaOH}}}_{\text{Converts to mmol of H}_2\text{PO}_4^-} \\
 &\times \underbrace{\frac{0.09699 \text{ g H}_2\text{PO}_4^-}{1 \text{ mmol H}_2\text{PO}_4^-}}_{\text{Converts to g of H}_2\text{PO}_4^-} \times \underbrace{\frac{100 \%}{\text{Sample Mass (g)}}}_{\text{Converts to \% w / w of H}_2\text{PO}_4^-}
 \end{aligned}$$

Experimental Percentage by Mass of the Dihydrogen Phosphate Ion (% w / w)
= _____ % w / w

The DATA TABLES AND REPORT Section Continues on the Next Page →

Name		Day		Start Time	
------	--	-----	--	------------	--

DATA TABLES AND REPORT (Cont.)

Table G: Gravimetric pH Titration of CO_3^{2-} Solution with 0.1000 M HCl Solution (Cont.)

Weighings of Substance B (In INK to Four Places After the Decimal)

Initial Mass (g)	Final Mass (g)	Sample Mass (g)

Data and Plotting: See the **Report Instruction R-13** on page 28 and the graphic below.

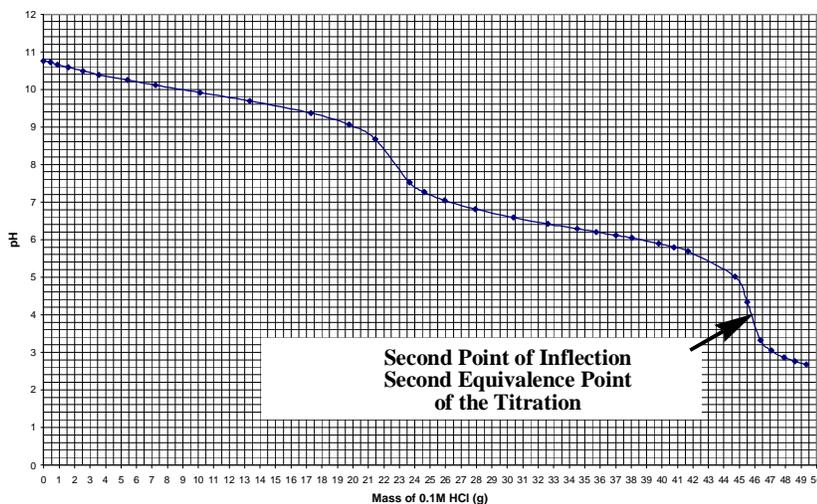
Estimated Mass of 0.1000 M HCl Solution Added at the **Second** Inflection Point (g)

= _____ g (To nearest 0.1 g)

Estimated pH at the **Second** Equivalence Point = _____ pH (To nearest 0.1 unit)

Attach your Data Table and Plot, showing how the values were obtained.

Gravimetric Titration of Carbonate Ion Solution with 0.1M HCl Solution



**Data Collected
And Plotted by
Monica Mascarenhas
(1BT 2007)**

The DATA TABLES AND REPORT Section Continues on the Next Page →

DATA TABLES AND REPORT (Cont.)

Bonus Questions Attach the Answers on a Separate Page. Show Work

1. Estimate the experimental percentage by mass (% w / w) of carbonate ion in the substance **B**. Show and explain your calculation method. (10 points)
2. Suggest which, if any, visual pH indicator substance(s) listed in **Harris** would be able to satisfactorily indicate the end-point of the H_2PO_4^- – NaOH titration. Explain your answer in words and graphically on your titration plot. See **Harris** for a full discussion. (5 points)
3. Suggest which, if any, visual pH indicator substance(s) listed in **Harris** would be able to satisfactorily indicate the **second** end-point of the CO_3^{2-} – HCl titration. Explain your answer in words and graphically on your titration plot. See **Harris** for a full discussion. (5 points)

Mark Sheet for Experiment 7

Total = 100 points

Category	Points
Pre-Laboratory Preparation	/ 15
Attendance: Punctuality, Diligence, Clean-Up.	/ 25
Data Acquisition and Recording In Ink /5 Proper Number of Digits /5 Results Initialed Weighings /5 pH /5 H_2PO_4^- Titration /5 CO_3^{2-} Titration /5	/ 30
Calculations Salt Concentration /3 Analytical Calculation /7	/ 10
Quality and Use of a Plot Quality of the Plots /10 Use of the $\text{H}_2\text{PO}_4^{2-}$ Plot /10	/ 20
Analytical Result for % w / w of Dihydrogen Phosphate Ion Result Correct Within: $\pm 3.0\% = 20$ $\pm 5.7\% = 16$ $\pm 10.8\% = 12$ $\pm 20.6\% = 8$ $\pm 39.1\% = 4$	/ 20
Bonus Section: Question 1 / 10 Question 2 / 5 Question 3 / 5	/ 20
Comments:	Total = /120