

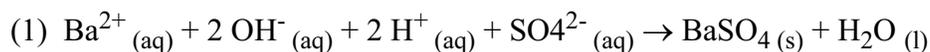
18 Conductometric Titration

Background

Titration is a method of determining the concentration of an unknown solution (the *analyte*) by reacting it completely with a standardized reagent that is a solution of known concentration (the *titrant*). The point at which all of the analyte is consumed is the *equivalence point*. The number of moles of analyte is calculated from the volume of reagent that is required to react with all of the analyte, the titrant concentration, and the reaction stoichiometry.

Previously, a pH electrode was used to measure the pH of the analyte and to determine when the equivalence point was reached. Another method of determining when the equivalence point is reached is by using a conductivity sensor to measure the conductivity of the analyte.

The conductivity of a solution is dependent on several factors, including the concentration of the solute, the degree of dissociation of the solute, the valence of the ion(s) present in the solution, the temperature, and the mobility of the ions in the solution. In this case, we will be titrating a solution of barium hydroxide, $\text{Ba}(\text{OH})_2$, with sulfuric acid, H_2SO_4 . Initially the $\text{Ba}(\text{OH})_2$ will be almost completely dissociated into Ba^{2+} and OH^- . With the addition of the H_2SO_4 the following reaction takes place:



The barium sulfate, BaSO_4 , is fairly insoluble and precipitates out of solution. The hydronium and hydroxide ions combine to form water. Neither of the reactant products contribute very much to the conductivity of the solution, thus as the titration takes place the conductivity will decrease. At the equivalence point, when enough H_2SO_4 has been added to react with the available $\text{Ba}(\text{OH})_2$, the conductance of the analyte is at its lowest point. Adding more titrant will cause the conductance of the analyte to increase again. Thus a plot of conductance vs. volume of H_2SO_4 added will result in a “V” shaped graph. We will take the curve fit for the line on either side of the equivalence point and solve for the intercept of the two lines to derive an exact value of H_2SO_4 used.

Purpose

In this lab, you will use a PASPORT Conductivity Sensor and Drop Counter to titrate a barium hydroxide solution of unknown concentration with a sulfuric acid solution of known concentration. You will use your data and the stoichiometry of the reaction to determine the concentration of the barium hydroxide solution.

Materials

PASCO & Other Equipment	
• PASPORT Xplorer GLX	• beaker, 150-mL (2)
• PASPORT Conductivity Sensor w/ 1x Conductivity electrode	• buret, 50-mL
• PASPORT Drop Counter	• graduated cylinder, 100-mL
• Right-angle clamp	• magnetic stirrer and stir bar
• Small Tripod Base & Rod	• pipet with bulb, 50-mL
• Buret Clamp	• wash bottle and waste container
• protective gear	

Consumables	
• Barium hydroxide ($\text{Ba}(\text{OH})_2$), unknown concentration, 50.0 mL	• Sulfuric acid (H_2SO_4), 0.02 M, approximately 60 mL
• Water, distilled, 100mL	

Safety Precautions

- Always work in an area that is well ventilated.
- Follow all directions for using the equipment.
- Wear safety glasses, gloves, goggles and protective clothing.
- Do not pipette by mouth.

Pre-Lab Questions

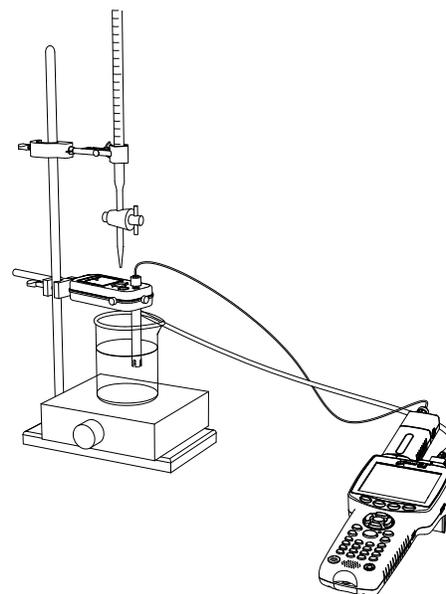
- 1) Sketch the expected appearance of the conductance vs. volume graph.
- 2) Indicate on the graph when the equivalence point is reached.

Procedure**Interface Setup**

- 1) Connect the Conductivity electrode to the Conductivity Sensor. Connect the Conductivity Sensor to Port #1 on the GLX
- 2) Connect the Drop Counter to Port #2 on the GLX.
- 3) Open the GLX file *18_Cond_Titration.glx*.

Equipment Setup

- 1) Set up the equipment as shown in the picture.
 - Use a right-angle clamp to attach the Drop Counter to the support rod. Above the Drop Counter, use a buret or three-finger clamp to affix a buret to the support rod. Ensure the buret valve is closed.
 - Place a magnetic stirrer at the base of the setup. Place the beaker you will be using for the titration on the stirrer.
 - Lower the Drop Counter until it rests just above the beaker. Then lower the buret so that the valve is about one inch above the Drop Counter opening.
- 2) Use a buret clamp to attach a 50 mL buret to the support rod with the tip about 1 inch above the opening in the drop counter. Ensure the buret stopcock is closed.
- 3) Fill the buret.
 - Remove the beaker from the magnetic stirrer and replace with a waste beaker.
 - Rinse your buret with 1-2 mL of titrant. Discard as directed. Then, fill your buret with ~50 mL of titrant, making sure not to fill past the top volume gradation. Drain a small amount of titrant through the drop counter into the waste beaker to remove any air in the tip of the buret and then practice adjusting the stopcock so that the titrant goes through the drop counter in distinguishable drops that fall at about 1 second intervals. Close the stopcock.
- 4) Read the initial volume of titrant in the buret to 0.01 mL. Record the volume and actual concentration of the 0.02 M H_2SO_4 in your data table.
- 5) The Conductivity Sensor is normally calibrated for the 10x electrode. Using the 1x electrode (necessary for the low conductivity values found in this experiment) will cause the conductivity values to read ten times higher than the actual value. In this experiment, however, the actual conductivity values are not important and the Conductivity Sensor does not require calibration.
- 6) Prepare the sample. Use the 50 mL pipet to add 50.0 mL of the barium hydroxide solution to the beaker. Record the volume of the barium hydroxide solution that you added in the data table. Use the graduated cylinder to add an additional 50 mL of distilled water to the beaker.



CAUTION: Never pipet by mouth. Always use a pipet bulb or a pipet pump. Be careful when handling any acid or base solutions.

- 7) Place the sample beaker on the stir plate, under the Drop Counter.
- 8) Install the supplied vertical stir bar in the end of the Conductivity electrode and insert the Conductivity electrode into one of the holes of the Drop Counter. Turn on the stir plate and begin stirring at a slow to medium speed.
- 9) The level of the water should be sufficient to cover the flow-through holes on the sides of the Conductivity electrode. If the water level is below the holes of the Conductivity electrode, add more distilled water until the holes are submerged.
- 10) Carefully examine the holes on the side of the electrode and check if there are any air bubbles trapped in the tip of the electrode. If there are air bubbles trapped in the tip, shake or tap the electrode to dislodge the air bubbles.

Record Data

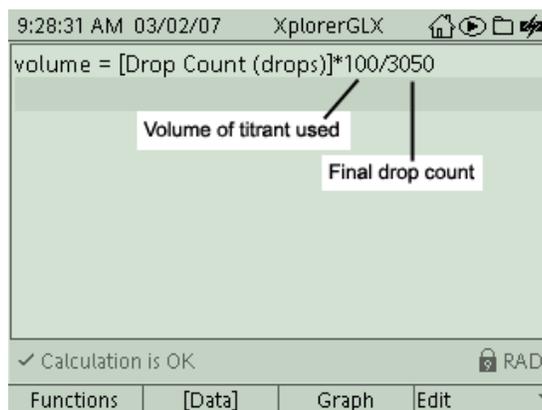
- 1) From the GLX Home screen, press $F1$ Graph. Setup the graph to display Conductivity versus Drop Count.
 - From the Home screen press $F1$ to open the **Graph Display**.
 - Press \checkmark to turn on the active fields.
 - Use the **Arrow Keys** to highlight the **Time** field (on the horizontal axis) and press \checkmark to open the data source menu.
 - Select **Drop Count (drops)** as the data source and press \checkmark again to accept the selection.
 - The Vertical axis should display conductivity.
- 2) Press \blacktriangleright to begin recording data. Turn the buret valve to release the H_2SO_4 at approximately one drop per second. Press the $F1$ Autoscale button to resize the titration graph and see the data.
- 3) Continue adding the H_2SO_4 titrant to the beaker until the conductivity value stops decreasing and starts to increase. At this point, continue to add between 5 -10 ml of H_2SO_4 and press \blacktriangleright again to end data recording.
- 4) Read the final volume of titrant in the buret and record the value in your data table. Calculate the total volume of titrant used.

- 5) Turn off the magnetic stirrer. Remove the Conductivity electrode from the solution. Rinse the Conductivity electrode in distilled water and dry the sensor gently.
- 6) Dispose of the solution in the beaker as instructed.

Analyze

Record calculations in your data table on the *Student Response Sheet* as you complete your analysis.

- 1) Read the total number of drops counted on the graph and record the final drop count in your data table.
- 2) In the GLX calculator screen ($F3$ from the Home screen), you will see a calculation similar to the one shown below. Modify the calculation using the values from your titration.



- 3) Return to the graph display. Change the data source for the horizontal axis of the graph from drops to the calculated “volume” using the procedure described in step 1 under Record Data above.
- 4) With the Graph displayed, press $F3$ Tools. Use the **Arrow keys** to select **Linear Fit** from the menu and press \checkmark . The GLX will perform a Linear Fit on the data and display the Slope and Y-intercept data for the curve. The Linear Fit takes into account the data from both sides of the equivalence point; however we need the fit data for one side or the other. You will need to select the data from the side that you wish to analyze.
- 5) With the Linear Fit selected, you will see a dashed-line Statistics Box. All the data within the Statistics Box will be used to perform the fit. Use the **Left and Right Arrow keys** to move the data cursor and select a fairly linear region of the graph that is before the equivalence point. If you need to move the start of the Statistics Box, press $F3$ Tools, use the **Arrow Keys** to select **Swap Cursors** and the press

- ①. Record the Slope and Y-intercept information that is displayed on the GLX screen for the data points before the equivalence point.
- 6) Similarly, use the **Arrow Keys** and **Swap Cursor** function to select a linear region of the graph that is after the equivalence point. Record the Slope and Y-intercept values shown on the GLX into the data table.
 - 7) Navigate to the GLX calculator by pressing \uparrow , then $F3$. The calculator displays the equation to find the equivalence point: $(b_2 - b_1) / (m_1 - m_2)$. Given the equation of a line ($m \cdot x + b$) where m is the Slope and b is the Y-intercept, enter the slope and Y-intercept data for before (m_1, b_1) and after (m_2, b_2) the equivalence point into the GLX calculator. The resulting answer is the volume of H_2SO_4 used at the equivalence point. Record this information to the Data Table.
 - 8) Use your recorded information to calculate the number of moles of H_2SO_4 used at the equivalence point.
 - 9) Use the stoichiometry given in Equation 1 to calculate the number of moles of $Ba(OH)_2$ and the molarity of the unknown sample.
 - 10) Follow your teacher's instructions regarding cleaning up your workspace.

Data Table

Item	Value
Volume of $Ba(OH)_2$	
Concentration of H_2SO_4	
Initial volume of titrant	
Final volume of titrant	
Final drop count	
Volume of titrant used	
Slope before equivalence point	
Y-intercept before equivalence point	
Slope after equivalence point	
Y-intercept after equivalence point	
Volume of H_2SO_4 used at equivalence point	
Moles of H_2SO_4 used at equivalence point	

Moles Ba(OH) ₂ in solution	
Concentration of Ba(OH) ₂ solution	

Analysis and Synthesis Questions

- 1) Derive the equation to find the equivalence point given the equation of two lines.
- 2) In this experiment, both the titrant and the analyte were conductive. What might the conductivity vs. volume curve look like if the analyte was not very conductive?

