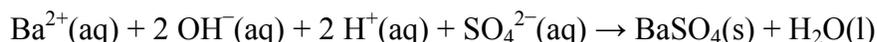


Conductimetric Titration and Gravimetric Determination of a Precipitate

In this experiment, you will monitor conductivity during the reaction between sulfuric acid, H_2SO_4 , and barium hydroxide, $\text{Ba}(\text{OH})_2$, in order to determine the equivalence point. From this information, you can find the concentration of the $\text{Ba}(\text{OH})_2$ solution. The reaction between sulfuric acid and barium hydroxide yields an insoluble product, barium sulfate, and water, as shown in the reaction equation below.



In this reaction, the total number of dissociated ions in solution is reduced dramatically during the reaction as a precipitate is formed. As 0.100 M H_2SO_4 is slowly added to $\text{Ba}(\text{OH})_2$ of unknown concentration, changes in the conductivity of the solution will be monitored using a Conductivity Probe. When the probe is placed in a solution that contains ions, and thus has the ability to conduct electricity, an electrical circuit is completed across the electrodes that are located on either side of the hole near the bottom of the probe body. This results in a conductivity value that can be read by the interface. The unit of conductivity used in this experiment is the microsiemens per cm, or $\mu\text{S}/\text{cm}$.

In addition, you will capture the precipitate, and measure its mass. You will have two methods, therefore, of calculating the molar concentration of a barium hydroxide solution that is titrated with a sulfuric acid solution of known concentration.

OBJECTIVES

In this experiment, you will

- Measure the conductivity of the reaction between sulfuric acid and barium hydroxide.
- Use conductivity values as a means of determining the equivalence point of the reaction.
- Measure the mass of a product of the reaction as a means of determining the equivalence point of the reaction gravimetrically.
- Calculate the molar concentration of a barium hydroxide solution.

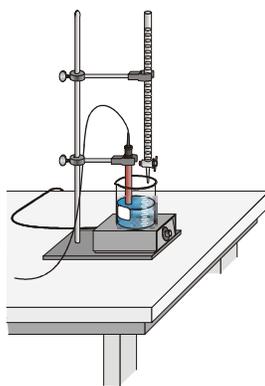


Figure 1

CHOOSING A METHOD

If you choose **Method 1**, you will conduct the titration in a conventional manner. You will deliver volumes of H_2SO_4 titrant from a buret. After titrant is added, and conductivity values have stabilized, you will manually enter the buret reading to store conductivity-volume data.

If you choose **Method 2**, you will use a Vernier Drop Counter to take volume readings. H_2SO_4 titrant is delivered drop by drop from the reagent reservoir through the Drop Counter slot. After the drop reacts with the reagent in the beaker, the volume of the drop is calculated, and a conductivity-volume data pair is stored.

MATERIALS

Materials for both Method 1 (buret) and Method 2 (Drop Counter)

TI-83 Plus or TI-84 Plus graphing calculator	magnetic stirrer
EasyData application	stirring bar or Vernier Microstirrer
data-collection interface	two ring stands
Vernier Conductivity Probe	ring stand ring
barium hydroxide, $\text{Ba}(\text{OH})_2$, solution	utility clamp
0.100 M sulfuric acid, H_2SO_4 , solution	filter paper
distilled water	filter funnel
two 250 mL beakers	balance, ± 0.01 gram accuracy (or better)
50 mL graduated cylinder	drying oven (optional)
10 mL pipet and pipet bulb or pump	

Materials required only for Method 1 (buret)

50 mL buret	buret clamp
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Materials required only for Method 2 (Drop Counter)

Vernier Drop Counter	100 mL beaker
60 mL reagent reservoir	10 mL graduated cylinder

METHOD 1 Measuring Volume Using a Buret

1. Obtain and wear goggles.
2. Use a pipet bulb (or pipet pump) to transfer 10.0 mL of the $\text{Ba}(\text{OH})_2$ solution into a 250 mL beaker. Add 50 mL of distilled water. **CAUTION:** *The barium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.*
3. Place the beaker on a magnetic stirrer and add a stirring bar. If no magnetic stirrer is available, you will stir with a stirring rod during the titration.
4. Turn on the calculator. Set the selector switch on the Conductivity Probe to the 0–20000 $\mu\text{S}/\text{cm}$ range. Connect the Conductivity Probe, data-collection interface, and graphing calculator.
5. Measure out approximately 60 mL of 0.100 M H_2SO_4 into a 250 mL beaker. Record the precise H_2SO_4 concentration in your data table. **CAUTION:** *H_2SO_4 is a strong acid, and should be handled with care.*

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6. Set up a ring stand, buret clamp, and 50.0 mL buret to conduct the titration (see Figure 1). Rinse and fill the buret with the H₂SO₄ solution.
7. Use a utility clamp to connect the Conductivity Probe to a ring stand, as shown in Figure 1. Position the Conductivity Probe in the Ba(OH)₂ solution and adjust its position so that it is not struck by the stirring bar.
8. Set up EasyData for data collection.
 - a. Start the EasyData application, if it is not already running.
 - b. Select **(File)** from the Main screen, and then select **New** to reset the application.
 - c. Select **(Setup)** from the Main screen, and then select **Events with Entry**.
9. Select **(Start)** to begin data collection. Conduct the titration carefully, as described below.
 - a. Before you have added any H₂SO₄ solution, select **(Keep)** and type in **0** as the buret volume in mL. Select **(OK)** to save the first data pair for this experiment.
 - b. Add 1.0 mL of 0.100 M H₂SO₄ to the beaker. When the conductivity value stabilizes, select **(Keep)** and enter the current buret reading. Select **(OK)** to save the second data pair for this experiment.
 - c. Continue adding 1.0 mL increments of H₂SO₄ solution, each time entering the buret reading, until the conductivity has dropped *below 100 μS/cm*.
 - d. After the conductivity has dropped below 100 μS/cm, add one 0.5 mL increment and enter the buret reading.
 - e. After this, use 2 drop increments (~0.1 mL) until the minimum conductivity has been reached at the equivalence point. Enter the volume after each 2 drop addition. When you have passed the equivalence point, continue using 2 drop increments until the conductivity is greater than 50 μS/cm again.
 - f. Now use 1.0 mL increments until the conductivity reaches about 1000 μS/cm, or 15 mL of H₂SO₄ solution have been added, whichever comes first.
10. Select **(Stop)** when you have finished collecting data.
11. Examine the data on the displayed graph to find the *equivalence point*; that is, the volume when the conductivity value reaches a minimum. As you move the cursor right or left on the displayed graph, the volume (X) and conductivity (Y) values of each data point are displayed above the graph. Record the H₂SO₄ volume of the point, with the minimum conductivity value, in your data table.
12. Store the data from the first trial.
 - a. Select **(Main)** to return to the Main screen.
 - b. Select **(File)**, and then select **Store Run**.
 - c. Select **(OK)** to store your latest data and overwrite the data in Lists 3 and 4 (L3 and L4).
13. Filter and measure the mass of the barium sulfate precipitate.
 - a. Use a hot plate to warm the beaker of mixture containing the BaSO₄ precipitate. Warm the solution to near boiling for about five minutes to help flocculate the particles.
 - b. While the mixture is heating, set up a ring stand and ring for the filter funnel. Measure and record the mass of a piece of fine grade-filter paper and set the paper in the funnel.

Calculator 16

- c. Allow the mixture to cool, and then filter it. The liquid need not be at room temperature to be filtered. Wash the precipitate out of the beaker with small amounts of distilled water, if necessary.
 - d. Dry the precipitate and filter paper in a drying oven for at least 15 minutes.
 - e. Cool the precipitate and filter paper to near room temperature. Measure and record the mass of the filter paper and precipitate.
 - f. Heat the precipitate again for five minutes, cool the precipitate, and weigh it.
 - g. Heat the precipitate a third time, for five more minutes, cool the precipitate and weigh it. If the masses of filter paper and precipitate are the same in the final two weighings, dispose of the filter paper as directed. If the final two weighings are not the same, check with your instructor to see if more drying time is needed.
14. Rinse the Conductivity Probe with distilled water in preparation for the second titration.
 15. Repeat the necessary steps to conduct a second titration. Conduct a third trial, if needed. Record the results in the data table. Print the graph of your final trial. Note: After selecting **(Start)** to begin data collection, select **(OK)** to start collecting data. Your stored data will not be overwritten.

METHOD 2 Measuring Volume Using a Drop Counter

1. Obtain and wear goggles.
2. Use a pipet bulb (or pipet pump) to transfer 10.0 mL of the $\text{Ba}(\text{OH})_2$ solution into a 250 mL beaker. Add 50 mL of distilled water. **CAUTION:** *The barium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.*
3. Set up the Conductivity Probe and Drop Counter.
 - a. Turn on the calculator and connect it to the data-collection interface.
 - b. Set the selector switch on the Conductivity Probe to the 0-20000 $\mu\text{S}/\text{cm}$ range.
 - c. Connect the Conductivity Probe to Channel 1 of the interface.
 - d. Lower the Drop Counter onto a ring stand as shown in Figure 2 and connect it to the DIG/SONIC 1 port of the interface.
4. Set up EasyData for data collection with a Drop Counter.
 - a. Start the EasyData application, if it is not already running.
 - b. Select **(File)** from the Main screen, and then select **New** to reset the application.
 - c. Select **(Setup)**, then select **Other Sensors**.
 - d. Select **DIG/SONIC1**, then select **(Next)**.
 - e. Select **Drop Counter**, then select **(Next)**.
5. Obtain the plastic 60 mL reagent reservoir. Close both valves by turning the handles to a horizontal position. Follow the steps below to set up the reagent reservoir for the titration.
 - a. Rinse the reagent reservoir with a few mL of the 0.100 M H_2SO_4 solution and pour the H_2SO_4 into an empty 250 mL beaker. **CAUTION:** *H_2SO_4 is a strong acid, and should be handled with care.*
 - b. Use a utility clamp to attach the reservoir to the ring stand.
 - c. Fill the reagent reservoir with slightly more than 60 mL of the 0.100 M H_2SO_4 solution.

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- d. Place the 250 mL beaker, which contains the rinse H_2SO_4 , beneath the tip of the reservoir.
- e. Drain a small amount of the H_2SO_4 solution into the 250 mL beaker so that it fills the reservoir's tip. To do this, turn both valve handles to the vertical position for a moment, then turn them both back to horizontal.
- f. Discard the drained H_2SO_4 solution in the 250 mL beaker as directed.

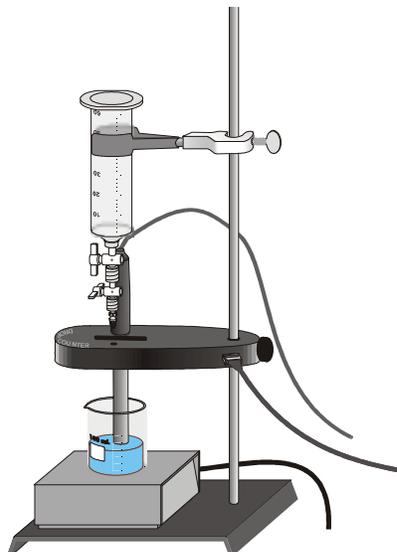


Figure 2

6. Calibrate the drops that will be delivered from the reagent reservoir.
 - a. Select **Setup** and then select **DIG1:Vol**.
 - b. Select **Calib** from the Sensor Setup screen.
 - If you have previously calibrated the drop size of your reagent reservoir and want to continue with the same drop size, select **Edit** and then select **Manual Entry...** Type the number of drops/mL, then select **Next**. Select **OK** twice, then proceed to Step 7.
 - If you want to perform a new calibration, continue with this step.
 - c. Select **Edit** and then select **Live...**
 - d. Place a 10 mL graduated cylinder directly below the slot on the Drop Counter, lining it up with the tip of the reagent reservoir.
 - e. Open the bottom valve on the reagent reservoir (vertical). Keep the top valve closed.
 - f. Slowly open the top valve of the reagent reservoir so that drops are released at a slow rate (~1 drop every 2 seconds).
 - g. Continue to release drops until there are between 9 and 10 mL of liquid in the graduated cylinder and close the bottom valve on the vessel to stop the drops.
 - h. Select **Next**.
 - i. Type the precise volume of liquid in the graduated cylinder in mL and select **OK**.
 - j. The number of drops/mL will be displayed. It may be helpful to record the value for that particular reagent reservoir for future titrations.
 - k. Select **OK** twice to return to the Main screen.
 - l. Discard the H_2SO_4 solution in the graduated cylinder as indicated by your instructor and set the graduated cylinder aside.

Calculator 16

7. Assemble the apparatus.
 - a. Place the magnetic stirrer on the base of the ring stand.
 - b. Insert the Conductivity Probe through the large hole in the Drop Counter.
 - c. Attach the Microstirrer to the bottom of the Conductivity Probe. Rotate the paddle wheel of the Microstirrer, and make sure that it does not touch the bottom of the Conductivity Probe.
 - d. Adjust the positions of the Drop Counter and reagent reservoir so they are both lined up with the center of the magnetic stirrer.
 - e. Lift up the Conductivity Probe, and slide the beaker containing the $\text{Ba}(\text{OH})_2$ solution onto the magnetic stirrer. Lower the Conductivity Probe into the beaker.
 - f. Adjust the position of the Drop Counter so that the Microstirrer on the Conductivity Probe is just touching the bottom of the beaker.
 - g. Adjust the reagent reservoir so its tip is just above the Drop Counter slot.
 - h. Turn on the magnetic stirrer so that the Microstirrer is stirring at a fast rate.
8. You are now ready to perform the titration.
 - a. Select $\overline{\text{Start}}$. No data will be collected until the first drop goes through the Drop Counter slot.
 - b. Fully open the bottom valve—the top valve should still be adjusted so drops are released at a rate of about 1 drop every 2 seconds. When the first drop passes through the Drop Counter slot, check the data table to see that the first data pair was recorded.
9. Observe that the conductivity readings decrease gradually. Continue watching your graph to see when the conductivity begins to increase; this will be the equivalence point of the reaction. When this increase in conductivity occurs, let the titration proceed for several more milliliters of titrant, then select $\overline{\text{Stop}}$. Turn the bottom valve of the reagent reservoir to a closed (horizontal) position.
10. Examine the data on the displayed graph to find the *equivalence point*—that is, the volume when the conductivity value reaches a minimum. As you move the cursor right or left on the displayed graph, the volume (X) and conductivity (Y) values of each data point are displayed above the graph. Record the H_2SO_4 volume of the point, with the minimum conductivity value, in your data table.
11. Leave EasyData and print a copy of the graph of conductivity vs. volume of H_2SO_4 for the first trial.
 - a. Select $\overline{\text{Main}}$ to return to the Main screen.
 - b. Select $\overline{\text{Quit}}$, then $\overline{\text{OK}}$ to leave EasyData.
 - c. Transfer the data to a computer, using Logger *Pro* software. Print a copy of your graph of conductivity vs. volume of H_2SO_4 .
12. Filter and measure the mass of the barium sulfate precipitate.
 - a. Use a hot plate to warm the beaker of mixture containing the BaSO_4 precipitate. Warm the solution to near boiling for about five minutes to help flocculate the particles.
 - b. While the mixture is heating, set up a ring stand and ring for the filter funnel. Measure and record the mass of a piece of fine-grade filter paper and set the paper in the funnel.
 - c. Allow the mixture to cool, and then filter it. The liquid need not be at room temperature to be filtered. Wash the precipitate out of the beaker with small amounts of distilled water, if necessary.

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- d. Dry the precipitate and filter paper in a drying oven for at least 15 minutes.
 - e. Cool the precipitate and filter paper to near room temperature. Measure and record the mass of the filter paper/precipitate.
 - f. Heat the precipitate again for five minutes, cool the precipitate, and weigh it.
 - g. Heat the precipitate a third time, for five more minutes, cool the precipitate and weigh it. If the masses of filter paper/precipitate are the same in the final two weighings, dispose of the filter paper as directed. If the final two weighings are not the same, check with your instructor to see if more drying time is needed.
13. Rinse the Conductivity Probe with distilled water in preparation for the second titration.
 14. Repeat the necessary steps to conduct a second titration. Conduct a third trial, if needed. Record the results in the data table. Print the graph of your final trial.

DATA TABLE

	Trial 1	Trial 2	Trial 3
Equivalence point (mL)			
Mass of filter paper + precipitate (g)			
Mass of filter paper (g)			
Mass of precipitate (g)			

Molarity of H ₂ SO ₄ (M)	
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DATA ANALYSIS

1. Use the titration results to calculate the moles of H₂SO₄ that were used to reach the equivalence point in each trial.

2. Use your titration results to calculate the molar concentration (molarity) of the Ba(OH)₂ solution using the molar amount of H₂SO₄ used in each trial.

Calculator 16

3. Convert the mass of the barium sulfate precipitate, formed in each trial, to moles.
4. Use the moles of BaSO_4 from 3 above to calculate the molarity of the $\text{Ba}(\text{OH})_2$ solution.
5. Compare the results of your calculations from 2 and 4 above with the actual molarity of the $\text{Ba}(\text{OH})_2$ solution. Which method of analysis, equivalence point or gravimetric determination, was more accurate in your experiment? Why?

TEACHER INFORMATION

Conductimetric Titration and Gravimetric Determination of a Precipitate

1. This experiment conforms to the guidelines for the 16th laboratory experiment listed in the College Board AP Chemistry guide (the Acorn book).
2. The student pages with complete instructions for data-collection using LabQuest App, Logger *Pro* (computers), EasyData or DataMate (calculators), DataPro (Palm handhelds), and a generic version can be found on the CD that accompanies this book. See *Appendix A* for more information.
3. Prepare 500 mL of 0.100 M sulfuric acid solution by adding 2.75 mL of concentrated H₂SO₄ solution to about 400 mL of distilled water and then adding distilled water to make a total volume of 500 mL.
4. Prepare 500 mL of the unknown barium hydroxide solution to an approximate concentration of 0.1 M by dissolving 16.67 g of Ba(OH)₂·8H₂O in 500 mL of distilled water. The solution will have a molarity that is significantly less than 0.1 M and the liquid will contain BaCO₃ precipitate from the dissolved CO₂ in the mix water. Filter the solution, to remove the carbonate impurities, before your students conduct this experiment.
5. In filtering the precipitate, your students should use the finest grain paper available. Alternately, the students can use a Gooch filter apparatus or a Büchner funnel with vacuum filtering.
6. If your students use a magnetic stirrer, make sure that they do not stir the mixture too vigorously. Rapid stirring may affect the conductivity readings and produce some anomalous data points during the titration.
7. Heating the reaction solution for 5–10 minutes before filtering off the precipitate will flocculate the barium sulfate and make it easier to collect the solid on filter paper.
8. Provide a container into which the students will dispose of their BaSO₄ and waste liquids. Please follow safe practices in handling and disposing of waste from this experiment.
9. The stored calibration for the Conductivity Probe works well for this experiment.
10. For best results, instruct your students to perform a new calibration on their Drop Counters. If time does not permit a new calibration, a substitute value of 28 drops/mL may be used. This value will provide good results. If students will not be calibrating their Drop Counters, instruct them follow the procedure for manually entering a calibration value and to enter a value of 28 for the drops/mL ratio.
11. If you are using a calculator and EasyData, there are several different combinations of equipment that will work for collecting conductivity data. The most common method, which works for both the TI-83 Plus and TI-84 Plus families of calculators, is to use a Conductivity Probe connected to a LabPro or a CBL 2. This is the equipment that must be used if you are collecting data with a calculator and a Drop Counter (Method 2 of the Procedure).

Experiment 16

The other combination of equipment, which uses the USB port on a TI-84 Plus calculator, is to collect data with a Conductivity Probe connected to an EasyLink. The EasyLink cannot be used with a Drop Counter because the Drop Counter is not supported by the EasyLink. For more information on EasyLink refer to *Appendix H*.

HAZARD ALERTS

Barium hydroxide: Toxic by ingestion. Hazard code: C—Somewhat hazardous.

Sulfuric acid: Severely corrosive to eyes, skin and other tissue; considerable heat of dilution with water; mixing with water may cause spraying and spattering. Solutions might best be made by immersing the mixing vessel in an ice bath. Extremely hazardous in contact with finely divided materials, carbides, chlorates, nitrates, and other combustible materials. Hazard code: A—Extremely hazardous.

The hazard information reference is: Flinn Scientific, Inc., *Chemical and Biological Catalog Reference Manual*, P.O. Box 219, Batavia, IL 60510, (800) 452-1261, www.flinnsci.com.

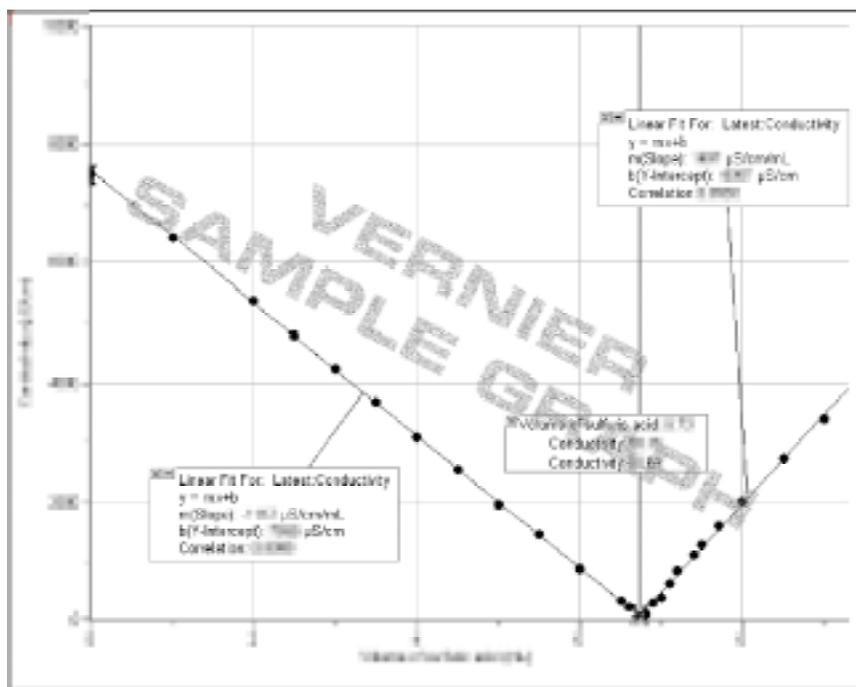
ANSWERS TO THE DATA ANALYSIS QUESTIONS

Answers have been removed from the online versions of Vernier curriculum material in order to prevent inappropriate student use. Graphs and data tables have also been obscured. Full answers and sample data are available in the print versions of these labs.

SAMPLE DATA

Equivalence point (mL)	x.xx
Mass of filter paper + precipitate (g)	x.xx
Mass of filter paper (g)	x.xx
Mass of precipitate (g)	x.xx

SAMPLE GRAPH



Plot of conductivity vs. volume of sulfuric acid