Conductimetric Titration and Gravimetric Determination of a Precipitate

In this experiment, you will monitor conductivity during the reaction between sulfuric acid, \( \text{H}_2\text{SO}_4 \), and barium hydroxide, \( \text{Ba(OH)}_2 \), in order to determine the equivalence point. From this information, you can find the concentration of the \( \text{Ba(OH)}_2 \) solution. The reaction between sulfuric acid and barium hydroxide yields an insoluble product, barium sulfate, and water, as shown in the reaction equation below.

\[
\text{Ba}^{2+}(\text{aq}) + 2 \text{OH}^-(\text{aq}) + 2 \text{H}^+(\text{aq}) + \text{SO}_4^{2-}(\text{aq}) \rightarrow \text{BaSO}_4(\text{s}) + \text{H}_2\text{O}(\text{l})
\]

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 \( \text{H}_2\text{SO}_4 \) is slowly added to \( \text{Ba(OH)}_2 \) 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 H₂SO₄ 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. H₂SO₄ 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)**
- LabQuest
- LabQuest App
- Vernier Conductivity Probe
- barium hydroxide, Ba(OH)₂, solution
- 0.100 M sulfuric acid, H₂SO₄, solution
- distilled water
- two 250 mL beakers
- 50 mL graduated cylinder
- 10 mL pipet and pipet bulb or pump
- magnetic stirrer
- stirring bar or Microstirrer
- two ring stands
- ring stand ring
- utility clamp
- filter paper
- filter funnel
- balance, ±0.01 gram accuracy (or better)
- drying oven (optional)

**Materials required only for Method 1 (buret)**
- 50 mL buret
- buret clamp

**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 Ba(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. Set the selector switch on the side of the Conductivity Probe to the 0–20000 µS/cm range. Connect the Conductivity Probe to LabQuest and choose New from the File menu. If you have an older sensor that does not auto-ID, manually set up the sensor.

5. Measure out approximately 60 mL of 0.100 M H₂SO₄ into a 250 mL beaker. Record the precise H₂SO₄ concentration in your data table. **CAUTION:** H₂SO₄ is a strong acid, and should be handled with care.

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. On the Meter screen, tap Mode. Change the data-collection mode to Events with Entry. Enter the Entry Label (Vol) and Unit (mL) and select OK.

9. Conduct the titration carefully, as described below.
   a. Start data collection
   b. Before you have added any H$_2$SO$_4$ solution, tap Keep. Enter 0, the volume in mL and select OK to save this data pair.
   c. Add 1.0 mL of 0.100 M H$_2$SO$_4$ to the beaker. When the conductivity value stabilizes, tap Keep. Enter 1 as the volume in drops and then select OK.
   d. Continue adding 1.0 mL increments of H$_2$SO$_4$ solution, each time entering the buret reading, until the conductivity has dropped below 100 µS/cm.
   e. After the conductivity has dropped below 100 µS/cm, add one 0.5 mL increment and enter the buret reading.
   f. 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.
   g. Now use 1.0 mL increments until the conductivity reaches about 1000 µS/cm, or 15 mL of H$_2$SO$_4$ solution have been added, whichever comes first.

10. Stop data collection to view a graph of conductivity vs. volume.

11. Examine the data on the displayed graph to find the equivalence point; that is, the volume when the conductivity value reaches a minimum. To examine the data pairs on the displayed graph, select any data point. Record the H$_2$SO$_4$ volume, at the point of minimum conductivity, in your data table.

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.
   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.

13. Rinse the Conductivity Probe with distilled water in preparation for the second titration.
14. Print the graph directly from LabQuest, if possible. Alternately, transfer the data to a computer, using Logger Pro software.

15. Repeat the necessary steps to conduct a second titration. Conduct a third trial, if needed. Record the results in the data table.

**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 Ba(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.*

![Figure 2](image)

3. Set up the Conductivity Probe and Drop Counter.
   a. Set the selector switch on the side of the Conductivity Probe to the 0–20000 µS/cm range and connect the Conductivity Probe to LabQuest.
   b. Lower the Drop Counter onto a ring stand and connect it to DIG 1.
   c. Choose New from the File menu. If you have an older Conductivity Probe that does not auto-ID, manually set up your Conductivity Probe. The Drop Counter is not an auto-ID sensor. Continue with this step to manually set up the Drop Counter.
   d. Choose Sensor Setup from the Sensor menu.
   e. Select Drop Counter from the D1 sensor list and select OK.
4. 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 H₂SO₄ solution and pour the H₂SO₄ into an empty 250 mL beaker. **CAUTION:** H₂SO₄ 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 H₂SO₄ solution.
   d. Place the 250 mL beaker, which contains the rinse H₂SO₄, beneath the tip of the reservoir.
   e. Drain a small amount of the H₂SO₄ 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 H₂SO₄ solution in the 250 mL beaker as directed.

5. Calibrate the Drop Counter so that a precise volume of titrant is recorded in units of milliliters.
   a. Choose Calibrate from the Sensors menu and select Drop Counter.
      - If you have previously calibrated the drop size of your reagent reservoir and want to continue with the same drop size, select Equation. Enter the values for the New Slope and the New Intercept. Select OK and proceed directly to Step 6.
      - If you want to perform a new calibration, select Start and continue with this step.
   b. Place a 10 mL graduated cylinder directly below the slot on the Drop Counter, lining it up with the tip of the reagent reservoir.
   c. Open the bottom valve on the reagent reservoir (vertical). Keep the top valve closed (horizontal).
   d. Slowly open the top valve of the reagent reservoir so that drops are released at a slow rate (~1 drop every two seconds). You should see the drops being counted on the screen.
   e. When the volume of H₂SO₄ solution in the graduated cylinder is between 9 and 10 mL, close the bottom valve of the reagent reservoir.
   f. Enter the precise volume of H₂SO₄ and tap Keep. Record the number of drops/mL for possible future use and select OK.
   g. Discard the H₂SO₄ solution in the graduated cylinder as indicated by your instructor and set the graduated cylinder aside.

6. 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 pH Sensor. 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 Ba(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.
7. You are now ready to perform the titration.
   a. Start data collection. 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 graph to see that the first data pair was recorded.
   c. 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.
   d. Stop data collection to view a graph of conductivity vs. volume.
   e. Turn the bottom valve of the reagent reservoir to a closed (horizontal) position.

8. Examine the data on the displayed graph to find the equivalent point; that is, the volume when the conductivity value reaches a minimum. To examine the data pairs on the displayed graph, select any data point. Record the $\text{H}_2\text{SO}_4$ volume, at the point of minimum conductivity, in your data table.

9. 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.
   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.

10. Rinse the Conductivity Probe with distilled water in preparation for the second titration.

11. Print the graph directly from LabQuest, if possible. Alternately, transfer the data to a computer, using Logger Pro software.

12. 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.
Conductimetric Titration and Gravimetric Determination of a Precipitate

DATA TABLE

<table>
<thead>
<tr>
<th></th>
<th>Trial 1</th>
<th>Trial 2</th>
<th>Trial 3</th>
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<tbody>
<tr>
<td>Equivalence point (mL)</td>
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<tr>
<td>Mass of filter paper + precipitate (g)</td>
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<tr>
<td>Mass of filter paper (g)</td>
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<tr>
<td>Mass of precipitate (g)</td>
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</tr>
<tr>
<td>Molarity of H₂SO₄ (M)</td>
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</tr>
</tbody>
</table>

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.

3. Convert the mass of the barium sulfate precipitate, formed in each trial, to moles.

4. Use the moles of BaSO₄ from 3 above to calculate the molarity of the Ba(OH)₂ solution.

5. Compare the results of your calculations from 2 and 4 above with the actual molarity of the Ba(OH)₂ solution. Which method of analysis, equivalence point or gravimetric determination, was more accurate in your experiment? Why?
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).
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


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.


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

<table>
<thead>
<tr>
<th>Sample Data</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Equivalence point (mL)</td>
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</tr>
<tr>
<td>Mass of filter paper + precipitate (g)</td>
<td>x.xx</td>
</tr>
<tr>
<td>Mass of filter paper (g)</td>
<td>x.xx</td>
</tr>
<tr>
<td>Mass of precipitate (g)</td>
<td>x.xx</td>
</tr>
</tbody>
</table>
SAMPLE GRAPH

Plot of conductivity vs. volume of sulfuric acid