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/cm, or \( \mu \text{S/cm} \).

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.
CHOOSING A METHOD

If you choose Method 1, you will conduct the titration in a conventional manner. You will deliver volumes of H$_2$SO$_4$ 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$_2$SO$_4$ 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)
- Vernier computer interface
- computer
- Vernier Conductivity Probe
- barium hydroxide, Ba(OH)$_2$, solution
- 0.100 M sulfuric acid, H$_2$SO$_4$, solution
- distilled water
- two 250 mL beakers
- 50 mL graduated cylinder
- 10 mL pipet and pipet bulb or pump
- hot plate
- magnetic stirrer
- stirring bar or Vernier Microstirrer
- two ring stands
- ring stand ring
- utility clamp
- filter paper
- filter funnel
- balance, ±0.01 gram accuracy (or better)
- drying oven

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. Connect a Conductivity Probe to Channel 1 of a Vernier computer interface. Connect the interface to the computer with the proper cable. Set the selector switch on the Conductivity Probe to the 0-20000 µS/cm range.

5. 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.
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6. 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. Use a buret clamp to attach a 50 mL buret to the ring stand as shown in Figure 1. Rinse and fill the buret with the H₂SO₄ solution.

7. Start the Logger Pro program on your computer. Open the file “16a Conductimetric” from the Advanced Chemistry with Vernier folder.

8. Click **Collect** to begin data collection. Conduct the titration carefully, as described below.
   a. Before adding H₂SO₄ titrant, monitor the displayed conductivity value (in µS/cm). Once the conductivity has stabilized, click **Keep**. In the edit box, type 0, the current buret reading in mL. Press ENTER to store 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, click **Keep**. In the edit box, type the current buret reading. Press ENTER.
   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.

9. Click **Stop** when you have finished collecting data.

10. Examine the data on the displayed graph to find the equivalence point; that is, the volume when the conductivity value reaches a minimum. Move the cursor across the graph to examine the data. As you move the examine line, the conductivity and volume values of each data point are displayed. Record the H₂SO₄ volume of the point with the minimum conductivity value in your data table.

11. Print a copy of the graph.

12. Filter and measure the mass of the barium sulfate precipitate.
   a. Use a hot plate to warm the 100 mL 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.
   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 trial.

14. Repeat the necessary steps to conduct a second trial. 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 10.00 mL of the Ba(OH)$_2$ solution into a 100 mL beaker. Add 40 mL of distilled water. **CAUTION:** The barium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.

3. Connect the Drop Counter to DIG/SONIC 1 of the Vernier computer interface and lower it onto the ring stand (see Figure 2).

4. Connect the Conductivity Sensor to Channel 1 of the interface. Set the selector switch on the Conductivity Probe to the 0-20000 range. Connect the interface to the computer with the proper cable.

5. Start the Logger Pro program on your computer. Open the file “16b Conductimetric (Drop)” from the Advanced Chemistry with Vernier folder.

6. Measure out approximately 60 mL of 0.100 M H$_2$SO$_4$ into a 250 mL beaker. Record the precise H$_2$SO$_4$ concentration in your data table. **CAUTION:** H$_2$SO$_4$ is a strong acid, and should be handled with care.
7. 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.
   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.

8. Calibrate the drops that will be delivered from the reagent reservoir. **Note:** If you are using the stored calibration (28 drops per mL), then skip this step.
   a. From the Experiment menu, choose Calibrate > DIG 1: Drop Counter.
   b. Proceed by one of these two methods:
      - If you have previously calibrated the drop size of your reagent reservoir and want to continue with the same drop size, select the Manual button, enter the number of Drops/mL, and click [OK]. Then proceed directly to Step 9.
      - If you want to perform a new calibration, select the Automatic button, and continue with this step.
   c. Place a 10 mL graduated cylinder directly below the slot on the Drop Counter, lining it up with the tip of the reagent reservoir.
   d. Open the bottom valve on the reagent reservoir (vertical). Keep the top valve closed (horizontal).
   e. Click the Start button.
   f. 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 computer screen.
   g. When the volume of the H₂SO₄ solution in the graduated cylinder is between 9 and 10 mL, close the bottom valve of the reagent reservoir.
   h. Enter the precise volume of H₂SO₄ in the edit box. Record the number of Drops/mL displayed on the screen for possible future use.
   i. Click [OK]. Discard the H₂SO₄ solution in the graduated cylinder as directed and set the graduated cylinder aside.

9. Assemble the apparatus.
   a. Place the magnetic stirrer on the base of the ring stand.
   b. Insert the Conductivity Sensor through the large hole in the Drop Counter.
   c. Attach the Microstirrer to the bottom of the Conductivity Sensor. Rotate the paddle wheel of the Microstirrer, and make sure that it turns freely.
   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 Sensor, and slide the 100 mL beaker containing the Ba(OH)₂ solution (from Step 2) onto the magnetic stirrer. Lower the Conductivity Sensor into the beaker.
Computer 16

f. Adjust the position of the Drop Counter so that the Microstirrer on the Conductivity Sensor is just touching the bottom of the beaker.
g. Adjust the reagent reservoir so its tip is just above the Drop Counter slot.

10. Conduct the titration.
   a. Click [Collect] to begin monitoring conductivity. No data will be collected until the first drop goes through the Drop Counter slot. 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.
   b. Observe your graph; the conductivity will drop below 100 µS and then rise again. The titration curve will be V-shaped. After the conductivity reaches about 1000 µS, click [Stop]. Turn the bottom valve of the reagent reservoir to a closed (horizontal) position.

11. Examine the data on the displayed graph to find the *equivalence point*; that is, the volume when the conductivity value reaches a minimum. Move the cursor across the graph to examine the data. As you move the examine line, the conductivity and volume values of each data point are displayed. Record the H₂SO₄ volume of the point with the minimum conductivity value in your data table.

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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.

14. Rinse the Conductivity Sensor 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.
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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Molarity of H₂SO₄ (M)

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?
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