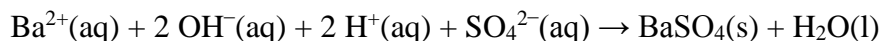


## Experiment 9

**Conductometric Titration & 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,  $\text{Ba}(\text{OH})_2$ , 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  $\text{H}_2\text{SO}_4$  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 micro Siemens 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.

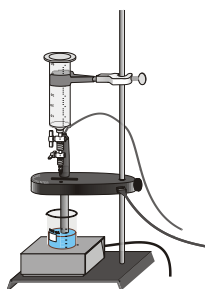


Figure 1

You will use a Vernier Drop Counter to take volume readings.  $\text{H}_2\text{SO}_4$  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 Drop Counter) Method

Vernier Drop Counter	100 mL beaker
60 mL reagent reservoir	10 mL graduated cylinder
LabQuest	magnetic stirrer
LabQuest App	stirring bar or Microstirrer
Vernier Conductivity Probe	two ring stands
barium hydroxide, $\text{Ba}(\text{OH})_2$ , solution	ring stand ring
0.100 M sulfuric acid, $\text{H}_2\text{SO}_4$ , solution	utility clamp
distilled water	filter paper
two 250 mL beakers	filter funnel
50 mL graduated cylinder	balance, $\pm 0.01$ gram accuracy (or better)
10 mL pipet and pipet bulb or pump	drying oven (optional)

## Measuring Volume Using a Drop Counter

1. Obtain and wear goggles.
2. Use a pipet bulb and Mohr pipet 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. Set the selector switch on the side of the Conductivity Probe to the 0–20000  $\mu\text{S}/\text{cm}$  range and connect the Conductivity Probe to LabQuest via either channel 1-4, located on the top of the LabQuest.
  - b. Lower the Drop Counter onto a ring stand and, using the cable, connect it to DIG 1 (located on the right-hand side of the LabQuest).
  - c. To set up LabQuest: Choose **New** from the File menu.
4. Obtain and assemble 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  $\text{H}_2\text{SO}_4$  solution and pour the  $\text{H}_2\text{SO}_4$  into an empty 250 mL beaker. **CAUTION:**  *$\text{H}_2\text{SO}_4$  is a strong acid, and should be handled with care.*
  - b. Use a utility clamp to attach the reservoir to the ring stand (see figure 1).
  - c. Fill the reagent reservoir with slightly more than 60 mL of the 0.100 M  $\text{H}_2\text{SO}_4$  solution.
  - d. Place the 250 mL beaker, which contains the rinse  $\text{H}_2\text{SO}_4$ , beneath the tip of the reservoir.
  - e. Drain a small amount of the  $\text{H}_2\text{SO}_4$  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  $\text{H}_2\text{SO}_4$  solution in the 250 mL beaker as directed.
5. Now you are ready to 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**.
    - To perform a new calibration, select **Calibrate Now** (top left side) and continue with step b.
    - NOTE: 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.

- 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  $\text{H}_2\text{SO}_4$  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  $\text{H}_2\text{SO}_4$  and select Stop. **Record** the number of drops/mL, in the box provided, for possible future use and select **OK**.
  - g. Discard the  $\text{H}_2\text{SO}_4$  solution in the graduated cylinder as indicated by your instructor and set the graduated cylinder aside.
6. Assembling the apparatus:
- a. Place the magnetic stirrer plate on the base of the ring stand.
  - b. Insert the Conductivity Probe through the large hole in the Drop Counter.
  - c. Place the magnetic stir bar in the bottom of the beaker 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 stir plate.
  - e. Lift up the Conductivity Probe, and slide the beaker containing the  $\text{Ba}(\text{OH})_2$  solution onto the magnetic stir plate. Lower the Conductivity Probe into the beaker.
  - f. Adjust the position of the Drop Counter so that 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 stir bar is stirring at a fast rate.
7. You are now ready to perform the titration.
- a. Directions for starting 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 (do not adjust the top valve since it has been calibrated in step 6). 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 (by pressing the green bottom left-hand “**keep**” icon) 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 *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  $\text{H}_2\text{SO}_4$  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  $\text{BaSO}_4$  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 weighing's, dispose of the filter paper as directed. If the final two weighing's 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.

## REPORT SHEET

Name: \_\_\_\_\_

## DATA TABLE I

	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 <sub>2</sub> SO <sub>4</sub> (M)	
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**DATA ANALYSIS** (If you need more space, provide your answers on the back page)

1. Use the titration results to calculate the moles of H<sub>2</sub>SO<sub>4</sub> 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)<sub>2</sub> solution using the molar amount of H<sub>2</sub>SO<sub>4</sub> used in each trial.
3. Convert the mass of the barium sulfate precipitate, formed in each trial, to moles.
4. Use the moles of BaSO<sub>4</sub> from 3 above to calculate the molarity of the Ba(OH)<sub>2</sub> solution.
5. Compare the results of your calculations from 2 and 4 above with the actual molarity of the Ba(OH)<sub>2</sub> solution. Which method of analysis, equivalence point or gravimetric determination, was more accurate in your experiment? Why?