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Catalog No. AP6188

Publication No. 6188

## Limiting Reagent Lab

### Formation of Barium Iodate

#### Introduction

If you have 12 hamburger buns, 20 pieces of cheese, and 3 hamburger patties, how many cheeseburgers can you make? This question may be simple, but it can help us learn about an important idea in chemistry—limiting reagents. Let's investigate the concept of limiting reagents with this lab activity.

#### Chemical Concepts

- Stoichiometry
- Limiting reagent
- Balanced chemical equations

#### Background

Most companies manufacture products made of other components. Having the correct quantity of each component is important to ensure efficient production. Ordering too many of one component creates unnecessary costs. If too few are available, production stops when the supply of that one component runs out. For example, no matter how many buns a fast food restaurant may have in stock, if the burgers run out, production stops. The burgers in this case could be called the *limiting reagent*.

Chemists often face similar situations. It is impossible for a chemist to make a certain amount of a desired compound if there is an insufficient quantity of any one of the required reactants. The balanced chemical equation is the chemist's "recipe." The coefficients in a balanced chemical equation tell us the whole number of moles of each reactant needed to complete the reaction in the correct proportions. In our example above, 1 hamburger bun + 1 hamburger patty + 1 piece of cheese → 1 cheeseburger. The whole numbers written in the equation are called coefficients. In chemical reactions, to ensure that the limiting reactant is completely used up (and the reaction "goes to completion"), most recipes call for more than the minimum amount of the other reactants. These reactants are referred to as being present "in excess."

In this experiment, insoluble barium iodate  $\text{Ba}(\text{IO}_3)_2$  will be prepared by mixing solutions of barium chloride and potassium iodate, according to Equation 1.



The solutions will be mixed in test tubes of uniform size, so that the amounts of  $\text{Ba}(\text{IO}_3)_2$  precipitate produced can be compared by measuring the heights of the solids in the tubes. Different amounts of each stock solution will be assigned and class results will be analyzed. Using stock solutions of known concentrations of barium chloride and potassium iodate allows small amounts of each chemical to be measured out quickly and accurately, in varying ratios.

After the amount of precipitate has been measured, you will determine which of the reactants—barium chloride or potassium iodate—is still present in the tube in unreacted form (the *excess reagent*) and which has been completely used up (the *limiting reagent*). To do this, the precipitate is allowed to fully settle. Some of the clear liquid above the precipitate (called the *supernatant liquid*) is then extracted with a pipet. This liquid can be tested for unreacted starting material using two different chemical tests.

Barium ions ( $\text{Ba}^{2+}$ ) react readily with sulfate ions ( $\text{SO}_4^{2-}$ ) to make insoluble  $\text{BaSO}_4$ . When drops of the clear supernatant liquid are added to a sample of sodium sulfate solution, a dense white precipitate of  $\text{BaSO}_4$  forms. This indicates that not all of the barium ions have been used up. In other words, barium ions are in excess and *iodate is the limiting reagent*.

Iodate ions ( $\text{IO}_3^{-}$ ) are reduced to free iodine ( $\text{I}_2$ ) by bisulfite ions ( $\text{HSO}_3^{-}$ ). The resulting iodine then reacts with dissolved starch to form a dark blue complex. If a blue color appears when the clear supernatant liquid is added to bisulfite/starch solution, this indicates that not all of the iodate ions have been used up. In other words, iodate ions are in excess and *barium is the limiting reagent*.

## Materials

|   |   |
|---|---|
| Barium chloride solution, $\text{BaCl}_2$ , 0.2 M, 65 mL            | Reaction plate, 24-well (for testing excess ions) |
| Potassium iodate solution, $\text{KIO}_3$ , 0.2 M, 65 mL            | Syringes, 10-mL, 2                                |
| Sodium bisulfite/starch indicator solution, 90 drops                | Test tubes, 16 × 100 mm, 5                        |
| Sodium sulfate solution, $\text{Na}_2\text{SO}_4$ , 0.1 M, 45 drops | Test tube rack                                    |
| Beral-type pipets, 5 (for extracting supernatant liquids)           | Lab Data Charts, Stages 1–3                       |
| Labeling tape or pen  | Wood sticks (for stirring), 5                     |
| Metric ruler, graduated to 1 mm                                     |   |

## Safety Precautions

*Barium chloride solution is toxic by ingestion. Sodium bisulfite solution is a severe skin and tissue irritant and is slightly toxic; it has a slight sulfur odor; keep tightly capped. Avoid contact with all body tissues. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Wash hands thoroughly with soap and water before leaving the laboratory.*

## Pre-Lab Activity

- Obtain two 10-mL syringes—label one syringe as  $\text{BaCl}_2$  and the other as  $\text{KIO}_3$ .
- Label five test tubes A–E. (*Note:* All five test tubes should be the exact same height and diameter.)
- Label five Beral-type pipets A–E.
- Obtain five wood sticks for stirring.

## Procedure

### STAGE 1. Constant Iodate Volume

#### Part 1. Formation of Barium Iodate

1. Fill the appropriately-labeled 10-mL syringe with 0.2 M barium chloride solution. Fill the other labeled syringe with 0.2-M potassium iodate solution. Prepare the syringes to deliver accurate volumes by removing any air from the tips.
2. Place the five labeled test tubes in a test tube rack.
3. Following the STAGE 1 Data Chart, add the assigned volumes of  $\text{BaCl}_2$  and  $\text{KIO}_3$  to test tubes A–E. (*Note:* Add the larger volume of solution second to ensure efficient mixing. For example, if 1 mL of  $\text{BaCl}_2$  solution is to be combined with 3 mL of  $\text{KIO}_3$  solution, add the  $\text{KIO}_3$  last.) Stir the contents of each test tube with a separate wood stick.
4. Once the reagents have been thoroughly mixed, allow the solid precipitates to settle for about 3–5 minutes. Tap the tubes very gently so the precipitate settles evenly.
5. After the precipitates in each of the five tubes have settled undisturbed, use a metric ruler to measure the height in millimeters of solid in each tube. Record the height of each precipitate in the STAGE 1 Data Chart.

#### Part 2. Testing the Supernatant Liquid for the Excess Reagent

6. Obtain a 24-well reaction plate.
7. Use a Beral-type pipet labeled A to extract a small amount of the clear liquid from the top of tube A. (*Note:* Be careful to avoid drawing up any precipitate into the dropper.)
8. Add 3 drops of the liquid from pipet A to wells 1A and 1B in the well plate.
9. **Test for Excess  $\text{Ba}^{2+}$ :** Add 3 drops of 0.1 M sodium sulfate solution to well 1A and make observations. A cloudy white precipitate indicates that there are still barium ions in the solution. Determine whether or not there is excess  $\text{Ba}^{2+}$  and record this result in the STAGE 1 Data Chart.

10. *Test for Excess  $\text{IO}_3^-$* : Add 5 drops of sodium bisulfite/starch indicator solution to well 1B and make observations. An initial yellow followed by a dark blue color indicates that there are still iodate ions in the solution. Determine whether or not there is excess  $\text{IO}_3^-$  and record this result in the STAGE 2 Data Chart.
11. Repeat both tests (Steps 9 and 10) using pipet B and the liquid from the top of tube B. Use wells 2A and 2B.
12. Repeat both tests (Steps 9 and 10) for tubes C–E.
13. Clean out the test tubes, well plates, and pipets by combining all rinse solutions in a barium waste container as provided by your instructor. Do not dump any solutions down the drain.
14. Discuss class results before going on to STAGE 2.

### STAGE 2. Constant Barium Volume

15. Perform Steps 1–13 for STAGE 2 of the lab using the STAGE 2 Data Chart. Discuss class results before going on to STAGE 3.

### STAGE 3. Changing Volumes of Iodate and Barium Ions

16. Perform Steps 1–13 for STAGE 3 of the lab, using the STAGE 3 Data Chart. Discuss class results.

## Data Analysis

Complete the following for each of the Stages 1–3. Show all work on a separate sheet of paper.

1. Using the volume and concentration (0.2 M) of each starting material, calculate the number of starting millimoles (mmol) of  $\text{Ba}^{2+}$  and  $\text{IO}_3^-$  that were combined. Record this value in the appropriate Data Chart.
2. Use the balanced chemical equation for the reaction of barium ions with iodate ions from the *Background* section. What is the mole ratio of each reactant and product?
3. Use the equation to calculate the number of millimoles of solid  $\text{Ba}(\text{IO}_3)_2$  precipitate that are expected to form in each tube (A–E). Note which material is in excess and which is the limiting reagent (LR). Record the expected millimoles of precipitate in the Data Chart. Also record which material is expected to be in excess and the amount of excess in mmol. Record the limiting reagent for each tube. [*Hint*: Review your data chart to determine which reagent was completely used up—the *limiting reagent*. Start with mmoles of that limiting reagent (from #1) to calculate the mmoles of precipitate expected from the balanced equation.]
4. Compare the calculated number of mmoles of precipitate expected (from #3) with the height ratios found in the tubes. Account for any patterns that are observed. Determine the point at which each chemical becomes the limiting reagent for the reaction.

# Limiting Reagent Lab Data Chart

STAGE 1. Constant Iodate Volume

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

| Test Tube | Starting Vol Ba <sup>2+</sup> (mL) | Starting Vol IO <sub>3</sub> <sup>-</sup> (mL) | Actual Height of ppt (mm) | Excess Ba <sup>2+</sup> or IO <sub>3</sub> <sup>-</sup> ? | Starting mmol Ba <sup>2+</sup> | Starting mmol IO <sub>3</sub> <sup>-</sup> | Expected mmol ppt | Expected Excess? (mmol) |
|-----------|------------------------------------|--|---------------------------|---|--------------------------------|--|-------------------|-------------------------|
| A         | 1                                  | 3  |                           |   |                                |  |                   |                         |
| B         | 3                                  | 3  |                           |   |                                |  |                   |                         |
| C         | 5                                  | 3  |                           |   |                                |  |                   |                         |
| D         | 7                                  | 3  |                           |   |                                |  |                   |                         |
| E         | 9                                  | 3  |                           |   |                                |  |                   |                         |

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

# Limiting Reagent Lab Data Chart

STAGE 2. Constant Barium Volume

| Test Tube | Starting Vol Ba <sup>2+</sup> (mL) | Starting Vol IO <sub>3</sub> <sup>-</sup> (mL) | Actual Height of ppt (mm) | Excess Ba <sup>2+</sup> or IO <sub>3</sub> <sup>-</sup> ? | Starting mmol Ba <sup>2+</sup> | Starting mmol IO <sub>3</sub> <sup>-</sup> | Expected mmol ppt | Expected Excess? (mmol) |
|-----------|------------------------------------|--|---------------------------|---|--------------------------------|--|-------------------|-------------------------|
| A         | 3                                  | 1  |                           |   |                                |  |                   |                         |
| B         | 3                                  | 3  |                           |   |                                |  |                   |                         |
| C         | 3                                  | 5  |                           |   |                                |  |                   |                         |
| D         | 3                                  | 7  |                           |   |                                |  |                   |                         |
| E         | 3                                  | 9  |                           |   |                                |  |                   |                         |

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

# Limiting Reagent Lab Data Chart

STAGE 3. Changing Volumes of Iodate and Barium Ions

| Test Tube | Starting Vol Ba <sup>2+</sup> (mL) | Starting Vol IO <sub>3</sub> <sup>-</sup> (mL) | Actual Height of ppt (mm) | Excess Ba <sup>2+</sup> or IO <sub>3</sub> <sup>-</sup> ? | Starting mmol Ba <sup>2+</sup> | Starting mmol IO <sub>3</sub> <sup>-</sup> | Expected mmol ppt | Expected Excess? (mmol) |
|-----------|------------------------------------|--|---------------------------|---|--------------------------------|--|-------------------|-------------------------|
| A         | 9                                  | 1  |                           |   |                                |  |                   |                         |
| B         | 7                                  | 3  |                           |   |                                |  |                   |                         |
| C         | 5                                  | 5  |                           |   |                                |  |                   |                         |
| D         | 3                                  | 7  |                           |   |                                |  |                   |                         |
| E         | 1                                  | 9  |                           |   |                                |  |                   |                         |