

Oscillating Clock: Briggs-Rauscher Reaction

Preparer's Version

Introduction

Oscillating reactions represent a fascinating class of chemical phenomena characterized by periodic changes in reactant concentrations and observable properties such as color. The study of oscillating reactions has garnered significant attention in the field of chemical kinetics due to their intricate dynamics, which challenge traditional rate laws and underscore the complexity of reaction mechanisms. The Briggs-Rauscher reaction stands as a prominent exemplar within this domain, capturing the imagination of chemists since its discovery. This reaction involves a set of reagents including hydrogen peroxide, potassium iodate, sulfuric acid, starch, malonic acid, and manganese(II) sulfate, and showcases striking color changes from blue to colorless and back again over successive cycles.

Safety Hazards

- Personal Protective Equipment
 - Safety glasses/goggles
 - Nitrile gloves
 - Chemical & flame retardant lab coat
 - Physical Hazards
 - Hydrogen peroxide may intensify or cause fire.
 - Potassium iodate may intensify or cause fire.
 - Sulfuric acid is corrosive to metal.
 - Cornstarch is a very finely ground powder, may form combustible dust concentrations in air.
 - Chemical Hazards
 - Hydrogen peroxide may cause severe skin burns and eye damage; harmful if swallowed or inhaled.
 - Potassium iodate may cause severe eye damage; harmful if swallowed.
 - Sulfuric acid may cause severe skin and eye damage.
 - Cornstarch may cause serious respiratory irritation or compromise if inhaled.
 - Malonic acid causes serious skin and respiratory irritation; may cause severe eye damage.
 - Manganese sulfate may cause severe eye damage; may cause damage to central nervous system through prolonged or repeated exposure.
- Iodine causes skin, eye, and respiratory irritation; causes damage to respiratory system and central nervous system after single exposure; causes damage to kidney, liver, thyroid, and blood after repeated exposure; harmful if swallowed.

Materials

- 3 liters deionized water
- 410 mL 30% Hydrogen peroxide
- 43 g Potassium iodate
- 4.3 mL conc. Sulfuric acid (36N/18M)
- 16 g Malonic acid
- 3.4 g Manganese(II) sulfate monohydrate
- 0.3 g soluble Starch
- Sodium thiosulfate (see Clean-Up Procedures)
- 3x 2-liter beakers, 1x 100 mL beaker, 1x 50 mL beaker
- Hot/Stir plate & magnetic stir bar
- Glass stir rod
- Parafilm

Safety Data Sheet(s)

- [Hydrogen peroxide](#)
- [Potassium iodate](#)
- [Sulfuric acid](#)
- [Cornstarch](#)
- [Malonic acid](#)
- [Manganese sulfate](#)
- [Iodine](#)
- [Sodium thiosulfate](#)



Procedure

1. To make Solution A (4M Hydrogen Peroxide):
 - a. Measure 400 mL deionized water and pour it into a 2-liter beaker.
 - b. Measure 410 mL 30% hydrogen peroxide and pour it into the beaker of water.
 - c. Dilute the solution by adding deionized water until the total volume reaches 1.0 liter.
2. To make Solution B (0.20M Potassium Iodate & 0.077M Sulfuric Acid):
 - a. Measure 800 mL deionized water and pour it into a 2-liter beaker.
 - b. Weigh 43 g potassium iodate and add it to the beaker of water.
 - c. Measure 4.3 mL concentrated (36N/18M) sulfuric acid and add it to the mixture.
 - d. Warm gently and stir the solution until the potassium iodate has fully dissolved.
 - e. Dilute the solution by adding deionized water until the total volume reaches 1.0 liter.
3. To make Solution C (0.15M Malonic Acid, 0.02M Manganese(II) Sulfate, & Starch):
 - a. Measure 500 mL deionized water and pour it into a 2-liter beaker.
 - b. Weigh 16 g malonic acid and add it to the beaker of water.
 - c. Weigh 3.4 g manganese(II) sulfate monohydrate and add it to the mixture.
 - d. In a separate 100mL beaker, bring 50 mL of deionized water to a boil.
 - e. Weigh 0.3 g soluble starch and mix with approximately 5 mL deionized water using the glass stir rod to form a slurry in the weigh boat.
 - f. Pour the slurry into the 100mL beaker of boiling water. Continue heating and stirring until the starch is fully dissolved (1-2 minutes). The solution may still be turbid/cloudy.
 - g. Pour the starch solution into the solution of malonic acid and manganese(II) sulfate.
 - h. Dilute the solution by adding deionized water until the total volume reaches 1.0 liter.
4. Measure equal volumes of Solutions A, B, and C depending on the scale of the demonstration.
5. Provide a piece of parafilm for the flask when the reaction reaches its endpoint.

Tips & Tricks

- Agitating the mixture manually or on a stir plate can make the color changes occur more rapidly.

Clean-Up Procedures

1. Keep the flask securely sealed with parafilm until it can be placed in a fume hood. *Do not remove the parafilm until the flask is inside a functioning fume hood.*
2. Wait until the reaction is no longer generating gas (no more bubbles in solution). Carefully remove the parafilm.
3. Using a metal scoopula, add a *small* amount of solid sodium thiosulfate to the flask. Allow the reaction to settle before adding more.
4. Continue adding small quantities of sodium thiosulfate until the flask turns clear (neutralized/quenched) or milky white (saturated with sodium thiosulfate).
 - a. *NOTE: The quenching process is extremely exothermic. Not only is this heat a hazard, but it also decreases the solubility of gasses in the solution. Add small amounts at a time to prevent overflowing.*
5. Pour waste into designated waste containers. You should have (1) quenched waste (large carboy, after the demonstration and quenching), (2) Solution A waste (small bottle, for labware used to make Solution A), (3) Solution B waste (small bottle, for labware used to make Solution B), and (4) Solution C waste (small bottle, for labware used to make Solution C).
6. Rinse the labware thoroughly with deionized water, emptying it into the carboy each time.
7. Thoroughly clean with laboratory soap.



Safety Notes:

- This reaction generates iodine vapors, which are highly toxic and have an extremely low exposure limit:
 - OSHA: The legal airborne permissible exposure limit (PEL) is 0.1 ppm, not to be exceeded at any time.
 - NIOSH: The recommended airborne exposure limit (REL) is 0.1 ppm, which should not be exceeded at any time.
- Iodine vapors appear as a vibrant pink/purple. It is normal for there to be a small amount of pink/purple staining on the parafilm as the reaction ceases, but it is NOT normal for the reaction to fume/release large quantities of iodine vapor.
- If the reaction does not proceed as anticipated – for example, does not go through the expected amount of color shifts/react for the expected duration of time – *proceed with caution*. Do not hesitate to contact EHS or a supervisor for assistance before quenching.
 - **February 24, 2024:** During an event, the reaction shifted colors only twice, indicating that there was a reactant limiting the reaction dramatically. It was immediately parafilmmed and moved to the fume hood until later in the day, when it could be quenched after the conclusion of the event. Upon attempting to quench the reaction in a fume hood, the extremely exothermic nature of the quenching process liberated excess amounts of iodine vapor rapidly and caused the flask to overflow. Personnel immediately shut the fume hood, evacuated the laboratory, and followed EHS Emergency Procedures by calling 911 for after-hours/weekend emergencies. Because the quenching was carried out inside of the fume hood, Hazmat professionals were able to determine that there was no iodine in the laboratory and therefore no continued risk. Demos Lab personnel were cleared for re-entry. The conclusion reached by the Demonstrations Coordinator, after consulting with Department of Chemistry faculty for second opinions and the original demonstration source material, was that Solution A was made incorrectly (over-diluted) or with hydrogen peroxide that had decomposed and was less active. This resulted in less hydrogen peroxide available in solution to react with elemental iodine and therefore an excess of iodine in solution after the reaction reached its endpoint. This incident showcased the importance of handling the waste of this reaction inside of a ventilated fume hood and the hazards of the reaction as a whole. It is crucial to communicate with UT Environmental Health & Safety if the reaction does not proceed as planned and there is cause for concern that there may be excess molecular iodine in solution that can be liberated as a gas if heated.