The Iodine Clock Reaction

EXPERIMENT 18B

It is very important for a chemist to understand the conditions that affect the rate of a chemical reaction. In chemical manufacturing processes, controlling the rate of a given reaction can make all the difference between an economical process and an uneconomical one. For instance, using catalysts is one method for getting reactions to speed up.

In the previous experiment (18A) you learned that the rate of a reaction is determined by several factors, namely temperature, the concentration of the reactants, and the surface area of the reactants (for a heterogeneous reaction). In this experiment you will carry out different reactions, but you will again investigate the effect of the concentration of reactants (Part I) and temperature (Part II) on reaction rate. In addition, in Part III you will examine the effect of the nature of the reactants, and in Part IV, the effect of a catalyst, on a reaction.

Part I involves a reaction that is sometimes called an *iodine clock reaction*. There are a number of different combinations of chemicals that give a reaction of this type. What happens, essentially, is that there are two different reactions: one in which iodine is produced (a slow reaction) and one in which the iodine produced in the first reaction is used up (a fast reaction). By carefully controlling the quantities of reactants, you can obtain a situation in which the reactant in the second reaction is used up first, allowing iodine to form at that point. At very low concentrations the iodine then combines with starch to suddenly give a deep blue-black color at a characteristic time for the conditions. Hence the term "iodine clock." The time taken after mixing until the point when the blue-black color appears is measured, and from this quantity the rate of the reaction can be determined. You will alter the conditions of concentration of reactants in Part I and temperature in Part II in order to determine their effect on reaction rate.

In Part III, you will observe how the nature of the reactants can affect the rate by reacting two different substances with the same reagent. Finally, in Part IV, you will observe the effect of adding a catalyst on the rate of a reaction, and you will look again at the effect of temperature.

OBJECTIVES

1. to observe and record the effect of changing the concentration of a reactant on the rate of a reaction
2. to observe and record the effect of changing the temperature of a system on the rate of a reaction
3. to observe and record the effect of the nature of the reactants on the rate of a reaction
4. to observe and record the effect of a catalyst on the rate of a reaction

MATERIALS

Apparatus

2 beakers (100 mL)
2 graduated cylinders (10 mL)
10 test tubes (18 mm × 150 mm)
thermometer
4 beakers (250 mL)
2 medicine droppers
ice
6 test tubes (13 mm × 100 mm)
lab apron
safety goggles

Reagents

Solution *A*: 0.02*M* KIO_3 (potassium iodate)
Solution *B*: 0.002*M* $NaHSO_3$ (sodium bisulfite) (also containing 4 g of starch and 12 mL of 1*M* H_2SO_4/L)
0.02*M* $KMnO_4$ (potassium permanganate)
0.1*M* $FeSO_4$ (iron(II) sulfate)
0.1*M* $Na_2C_2O_4$ (sodium oxalate)
0.1*M* $MnSO_4$ (manganese(II) sulfate)
1*M* H_2SO_4

PROCEDURE

Part I Effect of Concentration

1. Put on your lab apron and safety goggles.
2. Obtain approximately 60 mL of Solution *A* (0.02*M* KIO_3) and 90 mL of Solution *B* (0.002*M* $NaHSO_3$ containing H_2SO_4 and starch) in 100 mL beakers.
3. In a 10 mL graduated cylinder place 10.0 mL of Solution *A*, using a medicine dropper to obtain the volume as accurately as possible. Transfer the solution to an 18 mm × 150 mm test tube in a rack.
4. In the same manner measure out 10.0 mL of Solution *B* and transfer it to another test tube. Use a different graduated cylinder and medicine dropper than for Solution *A*, and keep the same ones for each solution for subsequent parts of the procedure.
5. In order to measure the time needed for the reaction to occur, you will need a watch or clock with a sweep second hand, or preferably, a digital watch with a stopwatch function. One partner must record the time while the other partner mixes the solutions. Mix the solutions in one of the two test tubes, and record the time from the instant they first mix.
6. Very quickly pour the solution back and forth between the two test tubes three times to make sure they are thoroughly mixed, then wait for the completion of the reaction.
7. Record the time at the instant the deep blue-black color first appears.
8. In order to study the effect of changing the concentration of Solution *A*, half the class will now be assigned the values 9.0 mL, 7.0 mL, 5.0 mL, and 3.0 mL, and the other half the values 8.0 mL, 6.0 mL, 4.0 mL, and 2.0 mL. In each instance measure out the volume in the graduated cylinder, and add enough water to make it up to the 10.0 mL mark. Then transfer each dilution of Solution *A* to a test tube and mix it with 10.0 mL of solution *B* as before. For each, record the time taken for the color to appear in Table 1 in your notebook.
9. Record your results on the board in order to get class averages.

Part II Effect of Temperature

1. In this part of the procedure you will keep the concentration constant, and vary the temperature, both above and below room temperature. In order that the observed reaction times lie in a suitable range, Solution *A* will be at only half the concentration of the original solution.
2. Make up 4 sets of each of solutions *A* and *B*, with 5.0 mL of Solution *A* and 5.0 mL of water in one set of 4 test tubes, and 10.0 mL of Solution *B* in the other 4 test tubes.
3. Make 4 water baths (in 250 mL beakers) at temperatures of 5°C, 15°C, 25°C, and 35°C or 10°C, 20°C, 30°C, and 40°C, depending on which set of temperatures your teacher assigns you. Use ice to obtain the temperatures below room temperature, and hot water from a tap or kettle to obtain the temperatures above room temperature. The beakers should be about two thirds full, so that the solutions in the test tubes are well beneath the water in the baths.
4. Place one test tube containing diluted Solution *A* and another containing Solution *B* in each water bath, and leave them for 10 min to allow them to adjust to the temperature required.
5. Try to maintain the temperatures in the water baths within 0.5°C of the temperatures assigned, to make comparisons with other groups in the class more meaningful.
6. When the temperatures are at the correct value, and the tubes have been in for enough time, mix each pair of solutions in one test tube by pouring them back and forth three times. Then place that test tube back in the water bath. In Table 2, record the time from the instant the solutions are mixed to the first appearance of the blue-black color. Do this for each pair of solutions at each temperature.
7. Record your results on the board in order to get class averages.

Part III Effect of the Nature of the Reactants

1. Place 3 mL of freshly prepared 0.1*M* $FeSO_4$ in a 13 mm × 100 mm test tube. Add 1 mL of 1*M* H_2SO_4, then add 5 drops of 0.02*M* $KMnO_4$, shaking after each drop.
2. Place 3 mL of 0.1*M* $Na_2C_2O_4$ in a 13 mm × 100 mm test tube. Add 1 mL of 1*M* H_2SO_4 and 5 drops of 0.02*M* $KMnO_4$.
3. Compare the lengths of time taken for the purple color to disappear in each test tube. (Leave them in the test-tube rack while you go on to Part IV.) Record your data in Table 3.

CAUTION: Potassium permanganate ($KMnO_4$) solution is a strong irritant and will stain skin and clothing. Wash any spills and splashes with plenty of water.

CAUTION: Sulfuric acid (H_2SO_4) is very corrosive. Do not get any on your skin, in your eyes, or on your clothing. Wash any spills and splashes with plenty of water, and call your teacher.

Part IV Effect of a Catalyst

1. In this part, you will be studying the same reaction as in Part III, Step 2, but using different temperatures and a catalyst. To each of four 13 mm × 100 mm test tubes add 3 mL of 0.1*M* $Na_2C_2O_4$ and 1 mL of 1*M* H_2SO_4.
2. Leave two of the test tubes in the rack at room temperature, and place the other two in a water bath at about 50°C.
3. To only one test tube at each temperature, add 3 drops of 0.1*M* $MnSO_4$ as a catalyst.
4. Then to all four tubes add 5 drops of 0.02*M* $KMnO_4$.
5. Record your data in Table 4, then compare the time taken to reach a colorless solution with and without the catalyst in each case. Also compare the times at room temperature with those at 50°C.
6. Clean up and put away all materials, following the instructions for reagent disposal.
7. Before leaving the laboratory, wash your hands thoroughly with soap and water; use a fingernail brush to clean under your fingernails.

REAGENT DISPOSAL

All solutions remaining after reaction may be safely rinsed down the sink with plenty of water. Your teacher will tell you what to do with the left-over original solutions.

POST LAB DISCUSSION

The blue-black color observed in Parts I and II occurs as a result of two separate reactions. Initially, the iodate ion, IO_3^-, reacts with the bisulfite ion, HSO_3^-, giving iodide ion, I^-, and sulfate ion, SO_4^{2-}, as follows:

$$IO_3^-(aq) + 3HSO_3^-(aq) \rightarrow I^-(aq) + 3SO_4^{2-}(aq) + 3H^+(aq)$$

The bisulfite ions are present in lower concentration and are, therefore, used up first. When this happens, the IO_3^- ions then react with I^- ions in the presence of H^+ ions to give molecular iodine, I_2:

$$IO_3^-(aq) + 5I^-(aq) + 6H^+(aq) \rightarrow 3I_2(aq) + 3H_2O(l)$$

In the presence of starch, iodine forms the intense blue-black color as a result of the iodine molecules being trapped in the long starch molecules. The appearance of this color indicates that the first reaction is complete and the second one has begun to take place.

The uncertainty involved in measuring the time taken for the reaction could easily be ±2 s unless you are very careful. Thus the interpretation of results will be more meaningful if the class averages at a particular concentration or temperature are considered. Measuring the time taken for a reaction to be completed is not the same as measuring its rate, but there is an inverse relation between them: rate is proportional to the reciprocal of time. Consequently, in interpreting the results you will calculate the rate in terms of reciprocal seconds (s^{-1}) and plot graphs of the rate against concentration or temperature.